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REMOVAL SUPPORT TEAM 3
EPA CONTRACT EP-S2-14-01

December 12, 2014

Mr. Ángel Rodríguez, On-Scene Coordinator
US Environmental Protection Agency
Caribbean Environmental Protection Division, Region II
City View Plaza Tower 2, Suite 7000
48 State Rd. 165, km 1.2, Guaynabo, Puerto Rico 00968-8069

EPA CONTRACT NO: EP-S2-14-01
TDD NO: TO-0001-0005
DOCUMENT CONTROL NO: RST3-01-F-0065
SUBJECT: SITE-SPECIFIC QUALITY ASSURANCE PROJECT PLAN – PUERTO RICO OLEFINS ASBESTOS SITE, PEÑUELAS, PUERTO RICO

Dear Mr. Rodríguez,

Enclosed please find the Site-Specific Quality Assurance Project Plan (QAPP) for the wipe, bulk asbestos, air, and microvacuum sampling events that have been and will be conducted at the Puerto Rico Olefins Asbestos Site located at Peñuelas, Puerto Rico beginning in July 2014.

If you have any questions, please do not hesitate to contact me at (787) 256-2501 or at (787) 354-2489.

Sincerely,

Weston Solutions, Inc.

Carlos L. Huertas
Environmental Engineer
Removal Support Team 3

Enclosure
cc: TDD File No.: TO-0001-0005

SITE-SPECIFIC QUALITY ASSURANCE PROJECT PLAN
PUERTO RICO OLEFINS ASBESTOS SITE
Peñuelas, Puerto Rico

NON-TIME CRITICAL

Prepared By:

Removal Support Team 3
Weston Solutions, Inc.
East Division
Santurce, Puerto Rico 00910

DC No.: RST 3-01-D-0061
TDD No.: TO-0001-0005
EPA Contract No.: EP-S2-14-01

November 2014

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ATTACHMENT B – Sampling SOPs

SOP # 2001: General Field Sampling Guidelines
SOP # 2008: General Air Sampling Guidelines
SOP # 2011: Chip, Wipe, and Sweep Sampling
SOP # 2012: Soil Sampling
SOP # 2015: Asbestos Air Sampling

ATTACHMENT C – Analytical Methods

LIST OF ACRONYMS

µm	micrometer
CA	corrective action
CAMP	Community Air Monitoring Plan
CARB	California Air Resources Board
CAS	Chemical Abstract Service
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	chain of custody
DCN	Document Control Number
DQI	data quality indicator
DQO	data quality objective
EDD	electronic data deliverable
EHS	Environmental Health and Safety
EPA	Environmental Protection Agency
ERT	Environmental Response Team
f/cc	fibers per cubic centimeter
HASP	Health and Safety Plan
HSO	Health and Safety Officer
ICS	Incident Command System
mm	millimeter
MS/MSD	matrix spike/matrix spike duplicate
NA	not applicable
NIOSH	National Institute for Occupational Safety and Health
NJDEP	New Jersey Department of Environmental Protection
NR	not required
NS	not specified
OSC	On-Scene Coordinator
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
PARCCS	Precision, Accuracy, Representativeness, Completeness, Comparability, Sensitivity
PCM	phase contrast microscopy
PLM	polarized light microscopy
QA	quality assurance
QAPP	Quality Assurance Project Plan
QAO	Quality Assurance Officer
QA/QC	quality assurance/quality control
QC	quality control
RST	Removal Support Team
SEDD	staged electronic data deliverable
SOP	standard operating practice
SPM	Site Project Manager
STR	sampling trip report

LIST OF ACRONYMS (Concluded)

TDD	Technical Direction Document
TO	task order
UFP	Uniform Federal Policy

CROSSWALK

The following table provides a "cross-walk" between the QAPP elements outlined in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP Manual), the necessary information, and the location of the information within the text document and corresponding QAPP Worksheet. Any QAPP elements and required information that are not applicable to the project are circled.

QAPP Element(s) and Corresponding Section(s) of UFP-QAPP Manual	Required Information	Crosswalk to QAPP Section	Crosswalk to QAPP Worksheet No.
Project Management and Objectives			
2.1 Title and Approval Page	- Title and Approval Page	Approval Page	1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	TOC Approval Page	 2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Approval Page	3 4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications - Special Personnel Training Requirements	2	5 6 7 8
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	1	 9 10
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria	3	11
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations	1 2	12 13

QAPP Element(s) and Corresponding Section(s) of UFP-QAPP Manual	Required Information	Crosswalk to QAPP Section	Crosswalk to QAPP Worksheet No.
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation - Project Schedule/Timeline	4	14 15 16
Measurement/Data Acquisition			
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/SOP Requirements - Analytical Methods/SOP Requirements - Field Quality Control Sample Summary - Sampling SOPs - Project Sampling SOP References - Field Equipment Calibration, Maintenance, Testing, and Inspection	5	17 18 19 20 21 22
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References - Analytical Instrument Calibration - Analytical Instrument and Equipment Maintenance, Testing, and Inspection	6	23 24 25
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	7	26 27

QAPP Element(s) and Corresponding Section(s) of UFP-QAPP Manual	Required Information	Crosswalk to QAPP Section	Crosswalk to QAPP Worksheet No.
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples - Screening/Confirmatory Analysis Decision Tree	5	NR
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records - Analytical Services - Data Management SOPs	6	29 30
Assessment/Oversight			
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments - Audit Checklists - Assessment Findings and Corrective Action Responses	8	31 32
4.2 QA Management Reports	- QA Management Reports		33 33
4.3 Final Project Report	- Final Report(s)		
Data Review			
5.1 Overview			
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process - Validation (Steps IIa and IIb) Process - Validation (Steps IIa and IIb) Summary - Usability Assessment	9	34 35 36 37

QAPP Worksheet #1: Title and Approval Page

Title: Site-Specific Quality Assurance Project Plan
Site Name/Project Name: Puerto Rico Olefins Asbestos Site
Site Location: Peñuelas, Puerto Rico
Revision Number: 01
Revision Date: Not Applicable

Weston Solutions, Inc.
Lead Organization

Carlos L. Huertas
Weston Solutions, Inc.
700 Europa St. Fernandez Juncos Corner Suite 206
Santurce, Puerto Rico, 00910
Email: carlos.huertas@westonsolutions.com
Preparer's Name and Organizational Affiliation

November 5, 2014
Preparation Date (Day/Month/Year)

Site Project Manager: _____
Signature

Carlos L. Huertas/Weston Solutions, Inc.
Printed Name/Organization/Date

QA Officer/Technical Reviewer: _____
Signature

Smita Sumbaly/Weston Solution, Inc.
Printed Name/Organization/Date

EPA, Region II On-Scene Coordinator (OSC): _____
Signature

Angel Rodriguez/EPA, Region II
Printed Name/Organization/Date

EPA, Region II Quality Assurance Officer (QAO): _____
Signature

Printed Name/Organization/Date

Document Control Number: RST 3-01-D-0061

QAPP Worksheet #2: QAPP Identifying Information

Site Name/Project Name: Puerto Rico Olefins Asbestos Site
Site Location: Peñuelas, Puerto Rico
Operable Unit: 00
Title: Site-Specific Quality Assurance Project Plan
Revision Number: 01
Revision Date: Not Applicable

1. **Identify guidance used to prepare QAPP:**
   Uniform Federal Policy for Quality Assurance Project Plans. Refer to EPA TEM Method CARB 435 Level B (soil), EPA Method 600/R-93/116 (bulk), ASTM D6480-05 TEM Method (wipe), NIOSH Methods 7400 and 7402 (air), and ASTM D5755-09 TEM Method (microvaccum).

2. **Identify regulatory program:** EPA, Region II

3. **Identify approval entity:** EPA, Region II

4. **Indicate whether the QAPP is a generic or a site-specific QAPP.**

5. **List dates of scoping sessions that were held:**

   7/11/2014, 7/29/2014, 7/31/14 and 9/29/2014

6. **List dates and titles of QAPP documents written for previous site work, if applicable:**

   Site-Specific Quality Assurance Project Plan, December 4, 2013.
   Site-Specific Quality Assurance Project Plan, March 17, 2014.

7. **List organizational partners (stakeholders) and connection with lead organization:** None

8. **List data users:** EPA, Region II (see Worksheet #4 for individuals)

9. **If any required QAPP elements and required information are not applicable to the project, then provide an explanation for their exclusion below:**

   Worksheet # 28 not required for asbestos analysis.

10. **Document Control Number:** RST 3-01-D-0061

QAPP Worksheet #3: Distribution List

[List those entities to which copies of the approved site-specific QAPP, subsequent QAPP revisions, addenda, and amendments are sent]

QAPP Recipient	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Angel Rodriguez	EPA, On-Scene Coordinator	EPA, Region II	(787) 671-8093	(787) 289-7104	rodriguez.angel@epa.epamail.gov	RST 3-01-D-0061
Geoffrey Garrison	EPA, On-Scene Coordinator	EPA. Region II	(787) 671-8101	(787) 289-7104	garrison.geoffrey@epa.epamail.gov	RST 3-01-D-0061
Carlos L. Huertas	Site Project Manager	Weston Solutions, Inc.	(787) 354-2489	(787) 256-2508	Carlos.Huertas@Westonsolutions.com	RST 3-01-D-0061
Smita Sumbaly	QA Officer	Weston Solutions, Inc., RST 3	(732) 585-4410	(732) 225-7037	S.Sumbaly@westonsolutions.com	RST 3-01-D-0061
Timothy Benton	HSO	Weston Solutions, Inc., RST 3	(732) 585-4425	(732) 225-7037	Tim.benton@westonsolutions.com	RST 3-01-D-0061
Site TDD File	RST 3 Site TDD File	Weston Solutions, Inc., RST 3	Not Applicable	Not Applicable	Not Applicable	-

QAPP Worksheet #4: Project Personnel Sign-Off Sheet

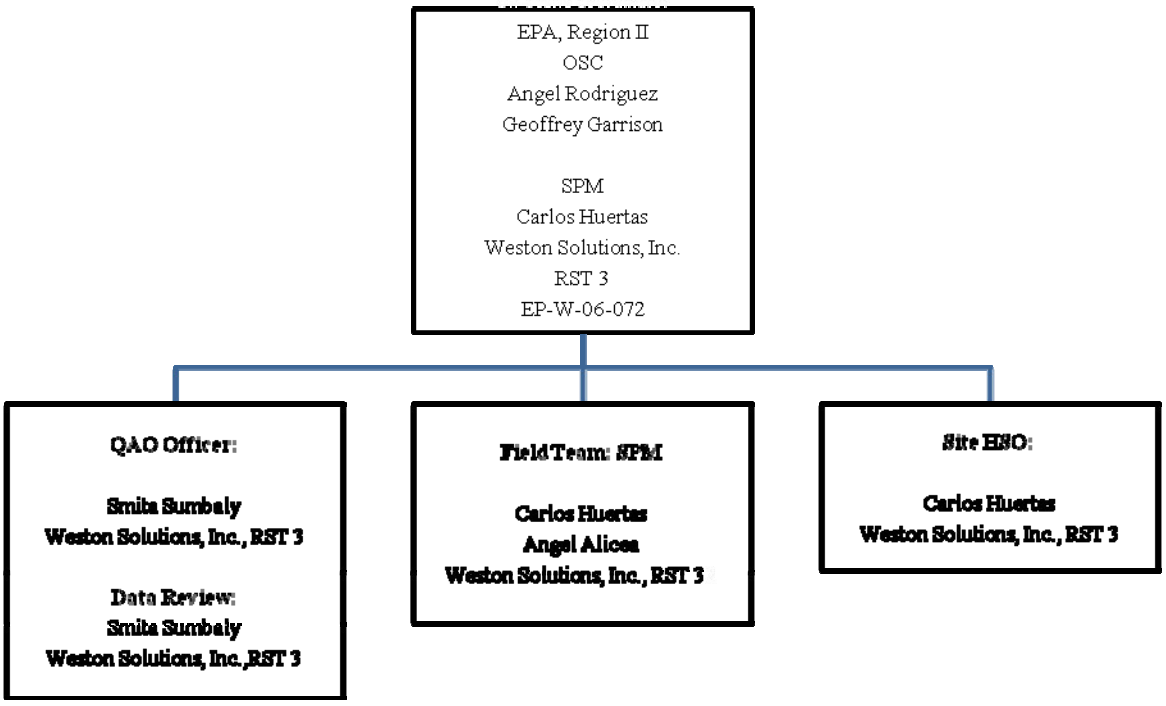
[Copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the site-specific QAPP and will perform the tasks as described; add additional sheets as required. Ask each organization to forward signed sheets to the central project file.]

Organization: Weston Solutions, Inc., RST 3

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Angel Rodriguez	EPA, Region II, On-Scene Coordinator	(787) 671-8093		11/5/14
Geoffrey Garrison	EPA, Region II, On-Scene Coordinator	(787) 671-8101		
Carlos L Huertas	Site Project Manager, RST 3	(787) 354-2489		11/5/14
Smita Sumbaly	QAO, RST 3	(732) 585-4410		11/05/14
Timothy Benton	HSO, RST 3	(732) 585-4425		
Erik Hadwin	Field Support, RST 3	(225) 773-9104		
Angel Alicea	Field Support, RST 3	(787) 365-9290		

QAPP Worksheet #5: Project Organizational Chart

Identify reporting relationship between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. Identify the organizations providing field sampling, on-site and off-site analysis, and data review services, including the names and telephone numbers of all project managers, project team members, and/or project contacts for each organization.



Acronyms:

EPA – U.S. Environmental Protection Agency
HSO – Health & Safety Officer
OSC – On-Scene Coordinator
QAO – Quality Assurance Officer
RST – Removal Support Team
SPM – Site Project Manager

QAPP Worksheet #6: Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure
Point of contact with EPA OSC	SPM, Weston Solutions, Inc., RST 3	Carlos Huertas SPM	(787) 354-2489	All technical, QA and decision-making matters in regard to the project (verbal, written or electronic)
Adjustments to QAPP	SPM, Weston Solutions, Inc., RST 3	Carlos Huertas SPM	(787) 354-2489	QAPP approval dialogue
Health and Safety On-Site Meeting	SPM, Weston Solutions, Inc., RST 3	Carlos Huertas SPM	(787) 354-2489	Explain/ review site hazards, personnel protective equipment, hospital location, etc.

OSC – On-Scene Coordinator
SPM – Site Project Manager

QAPP Worksheet #7: Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Angel Rodriguez	EPA OSC	EPA, Region II	All project coordination, direction and decision making.	NA
Geoffrey Garrison	EPA OSC	EPA, Region II	All project coordination, direction and decision making.	NA
Carlos Huertas	SPM, RST 3	Weston Solutions, Inc.	Implementing and executing the technical, QA and health and safety during sampling event and sample management.	5 years*
Erik Hadwin	Field Personnel, RST 3	Weston Solutions, Inc.	Sample collection and sample management	10 years*
Angel Alicea	Field Personnel, RST 3	Weston Solutions, Inc.	Sample collection and sample management	10 years*

OSC – On-Scene Coordinator
SPM – Site Project Manager
*All RST 3 members, including subcontractor's resumes are in possession of RST 3 Program Manager, EPA Project Officer, and Contracting officers.

QAPP Worksheet #8: Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[1]
[Specify location of training records and certificates for samplers]						
QAPP Training	This training is presented to all RST 3 personnel to introduce the provisions, requirements, and responsibilities detailed in the UFP QAPP. The training presents the relationship between the site-specific QAPPs, SOPs, work plans, and the Generic QAPP. QAPP refresher training will be presented to all employees following a major QAPP revision.	Weston Solutions, Inc., QAO	As needed	All RST 3 field personnel upon initial employment and as refresher training	Weston Solutions, Inc.	Weston Solutions, Inc., EHS Database
Health and Safety Training	Health and safety training will be provided to ensure compliance with Occupational Safety and Health Administration (OSHA) as established in 29 CFR 1910.120.	Weston Solutions, Inc., HSO	Yearly at a minimum	All Employees upon initial employment and as refresher training every year	Weston Solutions, Inc.	Weston Solutions, Inc., EHS Database
Others	Scribe, ICS 100 and 200, and Air Monitoring Equipment Trainings provided to all employees	Weston Solutions, Inc., QAO/Group Leader's	Upon initial employment and as needed			
	Dangerous Goods Shipping	Weston Solutions, Inc., HSO	Every 2 years			

All team members are trained in the concepts and procedures in recognizing opportunities for continual improvement, and the approaches required to improve procedures while maintaining conformance with legal, technical, and contractual obligations.

[1] All RST 3 members, including subcontractor's certifications are in possession of RST 3 HSO.

QAPP Worksheet #9: Project Scoping Session Participants Sheet

Site Name/Project Name: Puerto Rico Olefins Asbestos
Site Location: Peñuelas, Puerto Rico
Operable Unit: 00

Dates of Sessions: 7/11/2014, 7/29/2014 and 7/31/14
Scoping Session Purpose: To discuss questions, comments, and assumptions regarding technical issues involved with the project.

Name	Title	Affiliation	Phone #	E-mail Address	*Project Role
Angel Rodriguez / Geoffrey Garrison	EPA OSC	EPA, Region II	(787) 671-8093	Rodriguez.Angel@epamail.epa.gov	OSC
Carlos L Huertas	Site Project Manager	Weston Solutions, Inc.	(787) 354-2489	Carlos.Huertas@Westonsolutions.com	Project Management

Comments/Decisions: Removal Action of the Puerto Rico Olefins Asbestos Site (the Site) began on April 28, 2014. To confirm clean up of the Jorge Lucas Valdivieso School, Weston Solutions, Inc. Removal Support Team 3 (RST 3) has been task collect clearance wipe samples from classrooms selected by the On-Scene Coordinator (OSC). RST 3 will collect up to three wipe samples per classroom. Based upon analytical results, U.S. Environmental Protection Agency (EPA) will determine if the classroom is considered clean. RST 3 will also collected air, micro-vacuum, soil, bulk and wipe samples from six properties identified as impacted at the Tallaboa Encarnacion community. These samples will identify construction materials containing asbestos. RST 3 will collect additional multi-media samples for asbestos analysis, as needed and as directed by the EPA OSC.

Action Items: RST 3 submitted the Analytical Services Request Forms for analytical services on 7/11/2014 and 7/29/2014.

Consensus Decisions: EPA's OSC will select classrooms to be samples. Further sampling will be conducted as needed following EPA's review of sampling results. See the comments/decisions section of this report for a detailed summary of matrices and analysis.

QAPP Worksheet #9: Project Scoping Session Participants Sheet (Concluded)

Site Name/Project Name: Puerto Rico Olefins Asbestos
Site Location: Peñuelas, Puerto Rico
Operable Unit: 00

Dates of Sessions: 9/29/2014
Scoping Session Purpose: To discuss questions, comments, and assumptions regarding technical issues involved with the project.

Name	Title	Affiliation	Phone #	E-mail Address	*Project Role
Angel Rodriguez	EPA OSC	EPA, Region II	(787) 671-8093	Rodriguez.Angel@epamail.epa.gov	OSC
Carlos L Huertas	Site Project Manager	Weston Solutions, Inc.	(787) 354-2489	Carlos.Huertas@Westonsolutions.com	Project Management

Comments/Decisions: RST 3 will collect personal air samples at the PR Olefins facility to confirm asbestos concentration in air are not above the action level of 0.1 f/cc. Samples will be submitted for Asbestos to be analyzed by NIOSH 7400 and 7402 Methods.

Action Items: RST 3 submitted analytical request form for analytical services on July 11, 2014. 72 hours Turnaround Time (verbal) was requested for these samples.

Consensus Decisions: PPE level will be upgraded to Level C, if sample are above 0.1f/cc.

QAPP Worksheet #10: Problem Definition

PROBLEM DEFINITION

RST 3 has been tasked to provide multi-media sampling for asbestos during the Removal Action of the Puerto Rico Olefins Asbestos Site (the Site). RST 3 will collect air samples at the facility for Health and Safety purposes.

SITE HISTORY/CONDITIONS

The Puerto Rico Olefins Asbestos Site (the Site) is located at Road 127, KM 13.2, Tallaboa, Poniente Peñuelas, Puerto Rico. During a visual inspection, the U.S. Environmental Protection Agency (EPA) identified fugitive dust clouds migrating out of the facility during demolition activities conducted by HOMECA Inc. Beginning in 2010 and continuing until the present, an asbestos abatement occurred at the Site. Improper asbestos abatement techniques may have been used at the Site resulting in potential asbestos contamination throughout the Site and in residential neighborhoods downwind of the Site.

On November 21, 2013, as part of Phase I of the Removal Assessment, the EPA On-Scene Coordinator (OSC), EPA Air Program representative, and Weston Solutions, Inc., Removal Support Team 2 (RST 2) [currently the Removal Support Team 3 (RST 3)] mobilized to the Site to perform multi-media sampling. As directed by the EPA OSC, RST 2 collected five bulk samples, including one field duplicate, four soil samples, including one field duplicate, and 10 wipe samples, including one wipe blank. Bulk samples were collected and submitted for asbestos analysis via EPA 600/R-93/116 Method using Polarized Light Microscopy (PLM). Soil samples were collected and submitted for asbestos analysis via modified EPA 600/R-93/116 Method using Transmission Electron Microscopy (TEM) with California Air Resource Board (CARB) 435 prep. Wipe samples were collected and submitted for asbestos analysis via American Society for Testing Materials (ASTM) 6480-05 Method. Samples were collected from outside areas where suspected asbestos contamination may have occurred and inside areas where asbestos may have entered the building.

On December 13, 2013, as part of Phase I of the Removal Assessment, the EPA OSC and RST 2 remobilized to the Site to collect two additional bulk samples. The additional bulk samples were collected from two specific locations as directed by the EPA OSC. The two bulk samples were collected and submitted for asbestos analysis via EPA 600/R-93/116 Method using PLM. Samples were collected from outside areas where suspected asbestos contamination may have occurred.

Based on the validated analytical results of the samples collected as part of Phase I of the Removal Assessment, asbestos was detected in bulk samples ranging from non-detect to 40 percent (%) amosite and 20% chrysotile, in soil samples ranging from 3 amosite/chrysotile asbestos structures to 9 amosite/chrysotile asbestos structures, and in wipe samples ranging from 7,760 structures per square centimeter (str/cm^2) to 374,000 str/cm^2.

QAPP Worksheet #10: Problem Definition (Continued)

The two additional bulk samples collected on December 13, 2013 were both non-detect for asbestos. The wipe blank sample was non-detect for asbestos.

On December 4 and 5, 2013, as part of Phase II of the Removal Assessment, the EPA OSC and RST 2 mobilized to the Jorge Lucas Perez Valdivieso School, located approximately 0.25 miles south of the Site, to conduct air sampling activities within classrooms identified by the EPA OSC. The school is separated into two areas referred to by RST 2 as Area 1 and Area 2. The two separate areas are separated by a road way. As directed by the EPA OSC, RST 2 established three air sampling stations within eight of the schools classrooms (CR01 through CR08). Air samples were collected from each of the established air sampling stations within each classroom but per the request of the EPA OSC only one of the air samples from each of the classrooms was submitted for asbestos analysis.

On December 5, 2013, as part of Phase II of the Removal Assessment, RST 2 shipped 11 air samples, including two lot blanks and one field blank, to the EMSL Analytical, Inc. laboratory for asbestos analysis via Method ISO 10312 - International Standard for the Determination of Asbestos Fibers - Direct Transfer. Per the request of the EPA OSC, on December 13, 2013, RST 2 shipped the additional two air samples collected on December 4, 2013 from classroom CR01 to the EMSL Analytical, Inc. laboratory for asbestos analysis via Method ISO 10312.

Based on the validated analytical results of the samples collected as part of Phase II of the Removal Assessment, chrysotile asbestos was detected in eight of the 10 field air samples submitted for asbestos analysis. The total number of asbestos structures in the positive detections ranged between 2 and 25. The reported concentrations in the positive detections ranged between 0.0004 structures per cubic centimeter (s/cc) and 0.0032 s/cc.

On December 12 and 13, 2013, as part of Phase IIIA of the Removal Assessment, the EPA OSC and RST 2 mobilized to the Jorge Lucas Perez Valdivieso School to perform wipe sampling within the classrooms of the school. The areas identified in each classroom to be sampled were the entrance, near a window, and the dustiest area in the room. A total of 90 wipe samples, including five field blanks and one lot blank, were collected from 28 classrooms (CR01 through CR29, excluding CR27). Classroom CR27 and some other general areas of the school were no sampled due to the fact that the areas were not accessible or as directed by the EPA OSC. Wipe samples were collected and submitted for asbestos analysis via ASTM 6480-05 Method.

Based on the validated analytical results of the samples collected as part of Phase IIIA of the Removal Assessment, asbestos was detected in wipe samples ranging from non-detect to 363,000 str/cm^2. The wipe blank samples were non-detect for asbestos.

On December 17 through 19, 2013, as part of Phase IIIB of the Removal Assessment, the EPA OSC and RST 2 mobilized to the Tallaboa Encarnacion Community to perform wipe sampling on the exterior of several properties. As directed by the EPA OSC, the area identified to be

QAPP Worksheet #10: Problem Definition (Continued)

sampled had to be exposed to ambient air, but not exposed to rain or had not been cleaned recently. A total of 27 wipe samples, including two field blanks, were collected from 24 properties (P0005 through P0028). Wipe samples were collected and submitted for asbestos analysis via ASTM 6480-05 Method.

Based on the validated analytical results of the samples collected as part of Phase IIIB of the Removal Assessment, asbestos was detected in wipe samples from non-detect to 32,200,000 str/cm^2. The wipe blank samples were non-detect for asbestos.

On January 2 and 3, 2014, as part of Phase IIIC of the Removal Assessment, the EPA OSC, RST 2, and the Puerto Rico Environmental Quality Board (EQB) mobilized to background locations, selected by the EPA OSC, at different distances and directions from the Site. As directed by the EPA OSC, the area identified to be sampled had to be exposed to ambient air, but not exposed to rain or have not been cleaned recently. A total of 13 wipe samples, including one field blank, were collected from 12 properties (P0029 through P0040). Properties P0029 through P0032 were located over five miles northwest of the Site; properties P0033 through P0036 were located over one mile north of the Site; and properties P0037 through P0040 were located over two miles southeast of the Site. Wipe samples were collected and submitted for asbestos analysis via ASTM 6480-05 Method.

Based on the validated analytical results of the samples collected as part of Phase IIIC of the Removal Assessment, asbestos was detected in wipe samples ranging from non-detect to 160,000 str/cm^2. The wipe blank sample was non-detect for asbestos.

On January 9, 2014, as part of Phase IIID of the Removal Assessment, the EPA OSC and RST 2 mobilized to the Head Start Encarnacion School (property P0014) to perform wipe sampling activities within the school. As directed by the EPA OSC, RST 2 collected five wipe samples from inside the classroom. The areas identified in each classroom to be sampled were the entrance, near a window, the dustiest area in the room, and two other high use areas. A total of five wipe samples were collected from property P0014. Wipe samples were collected and submitted for asbestos analysis via ASTM 6480-05 Method.

Based on the validated analytical results of the samples collected as part of Phase IIID of the Removal Assessment, asbestos was detected in wipe samples ranging from 2,910 str/cm^2 to 41,700 str/cm^2.

From March 4 through 27, 2014, as part of Phase IV of the Removal Assessment, the EPA OSC and RST 2 mobilized to the Tallaboa Encarnación area in Peñuelas, Puerto Rico to perform indoor air and micro vac sampling activities at 32 residential/commercial properties (property P0008 is considered two separate properties) located within 0.25 miles of the Site.

QAPP Worksheet #10: Problem Definition (Continued)

Air samples were submitted for asbestos analysis via PCM Method (NIOSH 7400) and TEM Method (NIOSH 7402), and/or ISO Method 13794:199(E). Microvacuum samples were submitted for asbestos analysis via ASTM Method D-5755-09.

Based on the validated PCM analytical results of the air samples collected as part of Phase IV of the Removal Assessment, asbestos was detected in each property sampled at concentrations ranging from <0.001 fibers per cubic centimeter (F/cc) to 0.02 F/cc. Six properties sampled were not able to be run for PCM analysis due to the cassettes being overloaded with particulates.

Based on the validated TEM analytical results of the air samples collected as part of Phase IV of the Removal Assessment, chrysotile, anthophyllite, actinolite, or tremolite asbestos fibers were detected in 16 of the 32 properties sampled. The reported concentrations in the positive detections ranged between 0.00018 s/cc to 0.5299 s/cc. In addition, six properties (including the separated property P0008) contained asbestos concentrations which exceeded the Site-Specific Action Levels of 0.0009 s/cc for residential properties and 0.002 s/cc for commercial properties.

Based on the validated TEM analytical results of the micro vac samples collected as part of Phase IV of the Removal Assessment, chrysotile, actinolite, or amosite asbestos fibers were detected in 26 of the 32 properties sampled. The reported concentrations in the positive detections ranged between 231.3 s/cm^2 to 12,782,000.0 s/cm^2. In addition, 13 properties (including the separated property P0008) contained asbestos concentrations which exceeded the Site-Specific Action Levels of 5,000 s/cm^2.

In April 2014, the potentially responsible party (PRP) initiated Removal Actions at the Jorge Lucas Perez Valdivieso School and the Head Start Encarnacion School. The Removal Actions were completed in August 2014.

PROJECT DESCRIPTION

The objective of the sampling event is to confirm clearance of the classroom at the Jorge Lucas Perez Valdivieso school and to identify construction materials containing asbestos at the six properties identified on Phase IV of the Removal Assessment. RST 3 will collect personal air samples to at the PR Olefins facility to confirm asbestos concentration in air are not above the action level of 0.1 f/cc.

QAPP Worksheet #10: Problem Definition (Concluded)

PROJECT DECISION STATEMENTS

Sampling will be conducted by RST 3 to identify/confirm the presence of asbestos. The data will be used by EPA to determine if classrooms were cleaned properly and to identify construction materials containing asbestos. If air samples collected at the facility are above 0.1 f/cc, PPE Level D will be upgraded to Level C.

QAPP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statement

Overall project objectives include: Sampling will be conducted by RST 3 to identify/confirm the presence of asbestos. The data will be used by EPA to determine if classrooms were cleaned properly and to identify construction materials containing asbestos If air samples collected at the facility are above 0.1 f/cc, PPE Level D will upgrade to Level C.

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Who will use the data? Data will be used by the EPA OSC.

What will the data be used for? The data will be used by EPA to determine if classrooms were cleaned properly and to identify construction materials containing asbestos. Air samples at the facility will be used to determine appropriate PPE Level.

What types of data are needed?
Matrix:Bulk, Wipe, Air, and Microvacuum

Type of Data: Definitive confirmation

Analytical Techniques: Off-site laboratory analyses by the following methods: EPA Method 600/R-93/116 (bulk), ASTM D6480-05 TEM Method (wipe), NIOSH Methods 7400 and 7402 (air), and ASTM D5755-09 TEM Method (microvacuum).

Parameters: Asbestos

Type of sampling equipments: Plastic zip lock bags (all samples), Ghost wipes (wipe), 0.45 µm, 25 mm cellulose ester membrane cartridges (air and microvacuum), high-volume sampling pumps (air), low-volume sampling pumps (air and microvacuum), hammers, chisel and utility knife.

Access Agreement: Obtained by the EPA OSC.

Sampling locations: Various locations throughout the Site and off site locations.

How much data are needed? Approximately 10 bulk samples, 60 wipe samples, 40 air samples using ISO 10312 Method, 40 air samples using NIOSH Methods 7400 and 7402, and 40 microvacuum samples will be collected. The total number of samples will increase, as needed and determined by EPA.

How "good" does the data need to be in order to support the environmental decision?
Definitive data confirmation analytical objective has been requested. Definitive data will support an intermediate or preliminary decision and to identify/confirm the presence of asbestos on site.

Where, when, and how should the data be collected/generated? On-site and off-site sampling locations will be determined by the EPA OSC. Wipe samples will be collected during removal activities during the period of July to August 2014. Dates for multimedia samples to be collected at the six identified properties will be determined by EPA. Personal air samples, including blanks, will be collected at the PR Olefins facility starting on September 2014.

QAPP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statement (Concluded)

Who will collect and generate the data? The samples will be collected by RST 3. Samples will be analyzed by an RST 3-procured laboratory and validated by an RST 3 data validator.

How will the data be reported? All data will be reported by the assigned laboratories (Preliminary, Electronics, and Hard Copy format). The Site Project Manager will provide a Sampling Trip Report, Status Reports, Maps/Figures, Analytical Report, and Data Validation Report to the EPA OSC.

How will the data be archived? Electronic data deliverables will be archived in the scribe database. Non-CLP hard copy data will be archive in EPA's document control room.

QAPP Worksheet #12A: Measurement Performance Criteria Table
Asbestos-TEM – EPA Method 600/R-93/116

(UFP-QAPP Manuel Section 2.6.2)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQI), measurement performance criteria (MPC) and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

Matrix	Bulk
Analytical Group	Asbestos-TEM
Concentration Level	%

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP#2001	EPA Method 600/R-93/116	Negative Control (field)	Not Determined	Field Duplicate	S & A
		Accuracy (field)	No analyte > LOD	Field Blank	S & A
		Precision (laboratory)	Varies by lab and asbestos concentration	Laboratory Duplicate/Replicate	A
		Accuracy (laboratory)	Varies by lab and asbestos concentration	Standard Reference Sample	A
		Negative Control (laboratory)	No analyte > LOD	Method Blank	A

* Field Duplicate samples are not required for bulk samples.

QAPP Worksheet #12B: Measurement Performance Criteria Table
Asbestos-TEM – ASTM D6480-05 TEM Method

(UFP-QAPP Manuel Section 2.6.2)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQI), measurement performance criteria (MPC) and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

Matrix	Wipe
Analytical Group	Asbestos-TEM
Concentration Level	Asbestos structures/cm^2

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP#2011	ASTM D6480-05 TEM Method	Accuracy (field)	No analyte > LOD	Lot Blank	S & A
		Precision (laboratory)	Varies by lab and asbestos concentration	Laboratory Duplicate	A
		Accuracy (laboratory)	Varies by lab and asbestos concentration	Standard Reference Sample	A
		Negative Control (laboratory)	No analyte > LOD	Method Blank	A

QAPP Worksheet #12C: Measurement Performance Criteria Table
Asbestos-PCM/TEM – NIOSH Method 7400/7402

(UFP-QAPP Manual Section 2.6.2)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQI), measurement performance criteria (MPC) and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

Matrix	Air
Analytical Group	Asbestos-PCM/TEM
Concentration Level	f/cc -variable

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP#2015	NIOSH Method 7400(PCM) and 7402 (TEM)	Precision (field)*	Not Required	Not Required	S & A
		Negative Control (field)	No analyte > RL	Field/Lot Blank	S & A
		Precision (laboratory)	Varies by analyst and asbestos concentration	Replicate Analysis	A
		Accuracy (laboratory)	Varies by analyst and asbestos concentration	Daily reference Slide	A
		Negative Control (laboratory)	No analyte > RL	Method Blank	A

* Field Duplicate samples are not required for air samples. All air samples will be run for both PCM and TEM analyses.

QAPP Worksheet #12D: Measurement Performance Criteria Table
Asbestos-TEM Method – ASTM D 5755

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQI), measurement performance criteria (MPC) and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

Matrix	Surface Dust
Analytical Group	Asbestos-TEM
Concentration Level	structure/centimeter^2

Sampling Procedure	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Detailed in ASTM D5755-09	ASTM D 5755-09 (Microvac)	Precision (field)	Not determined at this time, Lab dependent	Not Required	S & A
		Negative Control (field)	< 3asb structures	Field Blank	S & A
		Precision (laboratory)	Not determined at this time	Laboratory Duplicate/ Replicate	A
		Accuracy (laboratory)	Not determined at this time	Reference Standard	A
		Negative Control (field)	< 3asb structures	Lab Blank	A
		Intra QC	As per laboratory QA manual	Intra Lab analysis-replicate	A
		Inter QC	As per laboratory QA manual	Iner Lab analysis-replicate	A

*Field duplicate samples are not required for surface dust samples

QAPP Worksheet #13: Secondary Data Criteria and Limitations Table

Any data needed for project implementation or decision making that are obtained from non-direct measurement sources such as computer databases, background information, technologies and methods, environmental indicator data, publications, photographs, topographical maps, literature files and historical data bases will be compared to the DQOs for the project to determine the acceptability of the data. Thus, for example, analytical data from historical surveys will be evaluated to determine whether they satisfy the validation criteria for the project and to determine whether sufficient data was provided to allow an appropriate validation to be done. If not, then a decision to conduct additional sampling for the site may be necessary.

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data May Be Used (if deemed usable during data assessment stage)	Limitations on Data Use
Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable

QAPP Worksheet #14: Summary of Project Tasks

Sampling Tasks: During the Removal activities RST 3 will confirm clearance of Jorge Lucas Perez School (P0002) classrooms. Up to 20 wipe samples will be collected from the classrooms selected by the OSC. RST 3 will collect up to 10 bulk samples, 40 wipe samples, 20 air samples and 40 microvacuum samples from the six properties identified as impacted. A total of 20 air personal samples will be collected while providing maintenance to the equipment at the PR Olefins Site. The total number of samples will increase, as needed and determined by EPA. A definitive data deliverable has been requested; therefore field duplicate samples will be collected, where appropriate.

Analysis Tasks:
Asbestos EPA 600/R-93/116 - Bulk
Asbestos ASTM D6480-05 TEM Method- Wipe
Asbestos NIOSH Methods 7400 and 7402 – Air
Asbestos ASTM D5755-09 TEM Method – Microvacuum

Quality Control Tasks: Field duplicate samples are not required for wipe, air and dust matrix. It may be collected for the bulk samples. One field blank will be collected for every 20 samples and one lot blank sample for every new lot of air cartridges used.

Data Management Tasks: The data collected for the sampling activities will be organized, analyzed, and summarized in status and trip reports and other deliverables (e.g., analytical reports, final reports) that will be submitted to the OSC according to the Project Schedule. The reports will be prepared by the project manager and include appropriate data quality assessment. Standard methods and references will be used as guidelines for data reduction and reporting.

Documentation and Records: Field notebook, sample labels, custody seals, chain of custody, sample logs, etc.

All sample documents will be completed legibly, in ink. Any corrections or revisions will be made by lining through the incorrect entry and by initialing the error.

The following deliverables will be provided under this project:

Trip Report: A trip report will be prepared to provide a detailed accounting of what occurred during the sampling mobilization. The trip report will be prepared within 2 weeks of the last day of the sampling mobilization. Information will be provided on time of major events, dates, and personnel on-site (including affiliations).

Maps/Figures: Maps depicting site layout, contaminant source areas, and sample locations will be included in the trip report, as appropriate.

QAPP Worksheet #14: Summary of Project Tasks (Concluded)

Field Logbook: The field logbook is essentially a descriptive notebook detailing site activities and observations so that an accurate account of field procedures can be reconstructed in the writer's absence. The field logbook will be bound and paginated. All entries will be dated and signed by the individuals making the entries, and should include (at a minimum) the following:

1. Site name and project number
2. Name(s) of personnel on-site
3. Dates and times of all entries (military time preferred)
4. Descriptions of all site activities, site entry and exit times
5. Noteworthy events and discussions
6. Weather conditions
7. Site observations
8. Sample and sample location identification and description*
9. Subcontractor information and names of on-site personnel
10. Date and time of sample collections, along with chain of custody information
11. Record of photographs
12. Site sketches

* The description of the sample location will be noted in such a manner as to allow the reader to reproduce the location in the field at a later date.

Sample Labels: Sample labels will clearly identify the particular sample, and should include the following:

1. Site/project number.
2. Sample identification number.
3. Sample collection date and time.
4. Designation of sample (grab or composite).
5. Sample preservation.
6. Analytical parameters.
7. Name of sampler.

Sample labels will be written in indelible ink and securely affixed to the sample container. Tie-on labels can be used if properly secured.

Custody Seals: Custody seals demonstrate that a sample container has not been tampered with or opened. The individual in possession of the sample(s) will sign and date the seal, affixing it in such a manner that the container cannot be opened without breaking the seal. The name of this individual, along with a description of the sample packaging, will be noted in the field logbook.

Assessment/Audit Tasks: No performance audit of field operations is anticipated at this time. If conducted, performance and systems audits will be in accordance with the project plan.

Data Review Tasks: All data will be validated by RST 3.

QAPP Worksheet #15A: Reference Limits and Evaluation Table

Matrix: Bulk

Analytical Group: Asbestos

Concentration Level: High

Analyte	CAS Number	Project Quantiation Limit (%)	Analytical Method – EPA 600/R-93/116 Quantitation Limits (%)
Asbestos, via the EPA 600/R-93/116 Method	NA	< 1%	1 %

NA = Not Applicable

QAPP Worksheet #15B: Reference Limits and Evaluation Table

Matrix: Wipe

Analytical Group: Asbestos

Concentration Level: High

Analyte	CAS Number	Project Quantiation Limit	*Analytical Method – ASTM D6480-05 TEM Method Quantitation Limits
Asbestos, via the ASTM D6480-05 TEM Method	NA	260 asbestos Structure/cm^2	260 asbestos Structure/CM^*

NA = Not Applicable

* Laboratory will attempt to reach 260 structures/cm^2. However, the reporting limit is based on volume filtered by the lab, number of grid openings analyzed, and amount of dust contained in the sample. Therefore, it can vary.

QAPP Worksheet #15C: Reference Limits and Evaluation Table

Matrix: Air
Analytical Group: Asbestos
Concentration Level: Low

Analyte	CAS Number	OSHA Exposure Limit (f/cc)	Project Quantitation Limit (f/cc)	Analytical Method Limit of Detection (f/cc)*	Achievable Laboratory Limit*
Asbestos, via NIOSH Method 7400/7402**(Personal air samples)	NA	0.1	0.005 f/cc	<0.01 f/cc	0.005 f/cc
Asbestos, via NIOSH Method 7400/7402**(Residential/Commercial)	NA	0.1	0.0004 f/cc	<0.01 f/cc	0.0004 f/cc

f/cc – fibers per cubic centimeter
NA – not applicable
* The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The laboratory will be able to achieve a detection limit of 0.0004 f/cc with a volume of 3,500 liters and by analyzing at least 46 grid openings (Residential / Commercial samples only).
** All samples will be run for both PCM and TEM analysis in order to achieve the detection limit.

QAPP Worksheet #15D: Reference Limits and Evaluation Table

Matrix: Microvacuum

Analytical Group: Asbestos

Concentration Level: High

Analyte	CAS Number	Project Quantiation Limit	Analytical Method – ASTM D5755-09 TEM Method Quantitation Limits
Asbestos, via the ASTM D5755-09 TEM Method	NA	260 asbestos structures/cm^2	Target is 1,000 asbestos Structure/cm^2 with a reporting limit of <3,000 structures/cm^2*

NA = Not Applicable
* Laboratory will attempt to reach 260 structures/cm^2. However, the reporting limit is based on volume filtered by the lab, number of grid openings analyzed, and amount of dust contained in the sample. Therefore, it can vary.

QAPP Worksheet #16: Project Schedule/Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Preparation of QAPP	RST 3 Contractor Site Project Manager	Prior to sampling date	11/5/2014	QAPP	11/5/2014
Review of QAPP	RST 3 Contractor QAO and/or Group Leader	Prior to sampling date	11/4/2014	Approved QAPP	11/5/2014
Preparation of Health and Safety Plan	RST 3 Contractor Site Project Manager	Prior to sampling date	7/10/2014	HASP	7/15/2014
Procurement of Field Equipment	RST 3 Contractor Site Project Manager and/or Equipment Officer	Prior to sampling date	NA	NA	NA
Laboratory Request	RST 3 Contractor Site Project Manager and/or QAO	Prior to sampling date	7/11/2014 and 7/29/2014	Non-CLP Request Form	NA
Field Reconnaissance/Access	RST 3 Contractor Site Project Manager; or EPA Region II OSC	NA	NA	NA	NA
Collection of Field Samples	RST 3 Contractor Site Project Manager	7/11/2014	12/31/14	NA	NA
Laboratory Electronic Data Received	RST 3-procured Laboratory	7/20/2014	24 hrs from sampling date	Preliminary Data	NA
Laboratory Package Received	RST 3-procured Laboratory	7/30/2014	2 weeks from sampling date	Laboratory Data Package	-
Validation of Laboratory Results	RST 3-procured Laboratory	7/30/2014 to 1/15/15	1/30/2015	Data Validation Report	1/15/15
Data Evaluation/ Preparation of Final Report	RST 3 Contractor Site Project Manager	1/15/15	2 weeks from validated data	Final Report	1/31/15

QAPP Worksheet #17: Sampling Design and Rationale

RST 3 has been tasked to collect clearance wipe samples from classrooms selected by EPA OSC from the Jorge Lucas Perez Valdivieso School. RST 3 will also collec air, bulk, microvacuum and surface wipe from six properties impacted at the Tallaboa Encarnacion community. If air samples collected at the facility are above 0.1 f/cc, PPE Level will be upgraded to Level C.

Further sampling will be conducted as needed following EPA's review of sampling results. This sampling design is based on information currently available and may be modified on-site in light of field-screening results and other acquired information.

Wipe Sampling: Wipe samples will be collected from throughout the Site and off site locations and be submitted for asbestos TEM analysis, via the ASTM D6480-05 method. Samples will be collected utilizing dedicated Ghost Wipe™ pre-moistened sterile sampling pads. Each sample will be collected from a 10cm X 10cm (100 sq. cm) area, using a dedicated disposable template for each sample. The marked surface area to be sampled will be wiped firmly vertically and then horizontally to ensure complete surface coverage for analysis. The wipe samples will be placed in poly bags; therefore; no equipment decontamination will be required. All wipe samples will be collected in accordance with EPA ERT SOP # 2011.

Bulk Sampling: Bulk asbestos sample collection procedures will be dependent upon the location of the suspected ACM. An approximately 3 to 4-inch section of the ACM will be cut from the underlying structure using a safety utility knife. The sample area will be re-wetted and the ACM will be carefully removed and placed into the wet re-sealable plastic bag. After sealing the suspected ACM in the plastic bag, the exterior of the bag will be wetted, wiped with a clean paper towel, and placed into a second re-sealable plastic bag. If a chisel is required to collect the sample, a new clean chisel will be used for each sample location. The used chisel will be removed of any contamination, but not will be used to collect samples in any other location. The blade of the knife will be disposed after each sample collection and replaced with a new one. Bulk asbestos samples will be submitted for asbestos analysis via EPA Method EPA/600/R-93/116, Method for the Determination of Asbestos in Bulk Building Materials. All bulk samples will be collected in accordance with EPA ERT SOP # 2001.

Air Sampling: All air samples will be collected using dedicated sampling equipment; (0.45 µm, 25 mm cellulose ester membrane cartridge and high-volume sampling pumps for asbestos) therefore, no equipment decontamination will be required. For NIOSH 7400 and 7402 Methods, approximately 3,500 liter volume will be collected at the rate of 9 to 11 liters/minute for 6-7 hours period of time to achieve 0.0004 f/cc reporting limit as determined by the EPA OSC. Sample pumps will be calibrated in a clean zone using a high-volume flow meter, and a clean sample cartridge (with cover) of the same type as that used during sample collection. The initial flow rate is established based on the target sample volume and duration of sample collection. All air samples will be collected in accordance with EPA ERT SOP # 2008 and 2015.

QAPP Worksheet #17: Sampling Design and Rationale (Concluded)

Personal Air Sampling: Low flow personal air samples will be collected using a GilAir Plus. Personal air samples will be submitted for NIOSH 7400 and 7402 Methods, approximately 500 liters volume will be collected at the rate of 2 to 3.0 liters/minute for 3-4 hours period of time to achieve 0.005 f/cc reporting limit as determined by the EPA OSC.

Sample pumps will be calibrated in a clean zone using a high-volume flow meter, and a clean sample cartridge (with cover) of the same type as that used during sample collection. The initial flow rate is established based on the target sample volume and duration of sample collection. All air samples will be collected in accordance with EPA ERT SOP # 2008 and 2015. It was determined that air samples will be collected to determine exposure on the CRZ while providing periodic maintenance to the DUSTTRAK™ installed on site. Personal Protective Equipment will be upgraded to Level C if sample results are above OSHA PEL of 0.1 f/cc.

Microvacuum Sampling: Microvacuum surface-dust samples will be collected from the interior of properties using a 0.45 µm, 25 mm cellulose ester membrane cartridge and a low-volume pump. A small piece of tubing, about two inches in length and cut at a 45° angle, will be placed at the end of the cartridge. Each sample will be collected from a 10cm X 10cm (100 sq. cm) area, using a dedicated disposable template for each sample. The marked surface area to be sampled will be vacuumed for at least two minutes until all visible dust has been removed. The cartridge and the two inch piece of tubing will be sent to a laboratory for asbestos analysis via the ASTM D5755-09 method. All microvacuum samples will be collected in accordance with EPA ERT SOP # 2011.

The following laboratories will provide the analyses indicated:

Lab Name/Location	Dates	Sample Type	Parameters
Batta Laboratories 6 Garfield way Newark ,DE, 19713 Attn: Neraa Batta	July through December 2014	Air, wipe, microvacuum and Bulk	Asbestos

TBD – To be determined
Refer to Worksheet #20 for QA/QC samples, sampling methods and SOP.

QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements Table

Matrix	Sampling Location(s)	Unit	Analytical Group(s)	Concentration Level	No. of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
Bulk	6 or greater	% Asbestos	Asbestos (EPA TEM CARB 435 Method, Level B)	High	1 per 20 samples	ERT SOPs #2011	Site Contamination Investigation
Wipe	10 or greater	Structure/Cm^2	Asbestos (ASTM D6480-05 TEM Method)	High	NR	ERT SOPs #2011	Site Contamination Investigation
Air	6 or greater	f/cc	Asbestos PCM – NIOSH 7400 and TEM – NIOSH 7402	Low	NR	ERT SOPs #2008 and 2015	Site Contamination Investigation/ / Health and Saferty
Microvacuum	6 or greater	Structure/Cm^2	Asbestos (ASTM D5755-09 TEM Method)	High	NR	ERT SOPs #2001	Site Contamination Investigation

The website for EPA-ERT SOPs is: http://www.ert.org/mainContent.asp?section=Products&subsection=List

QAPP Worksheet #19: Analytical SOP Requirements Table

Matrix	No. of Samples	Analytical Group [Lab Assignment]	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements	Maximum Holding Time (preparation/ analysis)
Bulk	10	Asbestos (EPA 600/R-93/116 Method)	High	EPA 600/R-93/116 Method)	NA	1 Poly Bag	NA	NA
Wipe	60	Asbestos (ASTM D6480-05 TEM Method)	High	ASTM D6480-05 TEM Method	NA	Ghost Wipe in 2 oz. jar (10 cm. by 10 cm.)	NA	NA
Air	20	Asbestos PCM – NIOSH 7400 Method and TEM – NIOSH 7402 Method	Low	NIOSH 7400 and 7402	3,500 L at flow rate 9-11 L/min	Air Sampling Cartridge with a 0.45um, 25 mm cellulose ester membrane filter, 1 per sample	NA	NA
	*20				500 L at flow rate 2-3 L/min			
Microvacuum	40	Asbestos (ASTM D5755-09 TEM Method)	High	ASTM D5755-09 TEM Method	NA	Air Sampling Cartridge with a 0.45um, 25 mm cellulose ester membrane filter, 1 per sample	NA	NA

*Personal air samples

QAPP Worksheet #20: Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs[1]	No. of Extra Volume Laboratory QC (e.g., MS/MSD) Samples	No. of Rinsate Blanks	No. of Lot and Field Blanks	Total No. of Samples to Lab
Bulk	Asbestos (EPA 600/R-93/116 Method)	High	Asbestos (EPA 600/R-93/116 Method)	10 or greater	1/20 samples	NR	NR	NR	10
Wipe	Asbestos (ASTM D6480-05 TEM Method)	High	Asbestos (ASTM D6480-05 TEM Method)	150 or greater	NR	NR	NR	1 Lot Blank per Lot; 1 Field Blank per 20 Samples	60
Air (Residential/ Commercial)	Asbestos (PCM NIOSH 7400 and TEM NIOSH 7402)	Low	Asbestos PCM – NIOSH 7400 Method and TEM – NIOSH 7402 Method	100 or greater	NR	NR	NR	1 Lot Blank per Lot; 1 Field Blank per 20 Samples	40
Air (Personal air samples)									
Micro-vacuum	Asbestos (ASTM D5755-09 TEM Method)	High	Asbestos (ASTM D5755-09 TEM Method)	100 or greater	NR	NR	NR	1 Lot Blank per Lot; 1 Field Blank per 20 Samples	40

NR – not required

QAPP Worksheet #21: Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
ERT SOP #2001	General Field Sampling Guidelines, August 11, 1994, Rev. # 0.0	EPA/OSWER/ERT	To be determined based on type of material sampled	N	--
ERT SOP #2008	General Air Sampling Guidelines, November 16, 1994, Rev. # 0.0	EPA/OSWER/ERT	To be determined based on type of material sampled	N	--
ERT SOP # 2011	Chip, Wipe, and Sweep Sampling, November 16, 1994, Rev. # 0.0	EPA/OSWER/ERT	Wipes	N	--
ERT SOP #2012	Soil Sampling, February 18, 2000, Rev. # 0.0	EPA/OSWER/ERT	Plastic Scoops	N	--
ERT SOP #2015	Asbestos Air Sampling, November 17, 1994, Rev. # 0.0	EPA/OSWER/ERT	Sampling pump, cellulose ester membrane filter cartridge	N	--

See Attachment B for SOPs
Note: The website for EPA-ERT SOPs is: www.ert.org/mainContent.asp?section=Products&subsection=List

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing/ Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
High Volume Air Sampling Pumps	Daily calibration	AC is primary power. Keep internal trickle-charged battery with an approximate life of 8 hours	Visually insect the unit	Yearly	Follow equipment instruction	Replace batteries or replace unit if not working correctly	Equipment vendor	#2015

QAPP Worksheet #23: Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
EPA Method 600/R-93/116	Method for the Determination of Asbestos in Bulk Building Materials, July 1993	Definitive	Bulk	Polarized light Microscopy (PLM), X-Ray Diffraction (XRD), and Transmission Electron Microscopy (TEM).	Non-RAS Laboratory (NVLAP Certified)	N
ASTM 6480	Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Surface Loading by Transmission Electron Microscopy, March 2005	Definitive	Wipe	Transmission Electron Microscopy (TEM)	Non-RAS Laboratory (NVLAP Certified)	N
NIOSH 7400	Asbestos and Other Fibers by PCM, August 1994	Definitive	Air	Phase Contrast Microscopy (PCM)	Non-RAS Laboratory (NVLAP Certified)	N
NIOSH 7402	Asbestos by TEM, August 1994	Definitive	Air	Transmission Electron Microscopy (TEM)	Non-RAS Laboratory (NVLAP Certified)	N
ASTM 5755	Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading	Definitive	Microvacuum	Transmission Electron Microscopy (TEM)	Non-RAS Laboratory (NVLAP Certified)	N

QAPP Worksheet #24: Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Microscope	EPA 600/R-93/116	The TEM should be aligned daily to achieve illumination and centered through the substance condenser and iris diaphragm.	As per instrument manufacture's recommended procedures.	Inspect the system, correct problem, re-calibrate, and re-analyze samples.	Non-CLP Laboratory Microscope Technician	EPA 600/R-93/116
Microscope	ASTM D6480-05 TEM Method	The TEM should be aligned daily to achieve illumination and centered through the substance condenser and iris diaphragm.	As per instrument manufacture's recommended procedures.	Inspect the system, correct problem, re-calibrate, and re-analyze samples.	Non-CLP Laboratory Microscope Technician	ASTM D6480-05 TEM Method
Microscope	NIOSH 7400	The TEM should be aligned daily to achieve illumination and centered through the substance condenser and iris diaphragm.	As per instrument manufacture's recommended procedures.	Inspect the system, correct problem, re-calibrate, and re-analyze samples.	Non-CLP Laboratory Microscope Technician	NIOSH 7400
Microscope	NIOSH 7402	The PCM should be aligned daily to achieve illumination and centered through the substance condenser and iris diaphragm.	As per instrument manufacture's recommended procedures.	Inspect the system, correct problem, re-calibrate, and re-analyze samples.	Non-CLP Laboratory Microscope Technician	NIOSH 7402
Microscope	ASTM D5755-09 TEM Method	The TEM should be aligned daily to achieve illumination and centered through the substance condenser and iris diaphragm.	As per instrument manufacture's recommended procedures.	Inspect the system, correct problem, re-calibrate, and re-analyze samples.	Non-CLP Laboratory Microscope Technician	ASTM D5755-09 TEM Method

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Microscope	See EPA 600/R-93/116; as per instrument manufacturer's recommendations	See EPA 600/R-93/116; as per instrument manufacturer's recommendations	See EPA 600/R-93/116; as per instrument manufacturer's recommendations	Acceptable re-calibration; See EPA 600/R-93/116	Inspect the system, correct problem, re-calibrate and/or reanalyze samples.	Non-CLP Laboratory Microscope Technician	EPA 600/R-93/116
Microscope	See ASTM D6480-05 TEM Method; as per instrument manufacturer's recommendations	See ASTM D6480-05 TEM Method; as per instrument manufacturer's recommendations	See ASTM D6480-05 TEM Method; as per instrument manufacturer's recommendations	Acceptable re-calibration; See ASTM D6480-05 TEM Method	Inspect the system, correct problem, re-calibrate and/or reanalyze samples.	Non-CLP Laboratory Microscope Technician	ASTM D6480-05 TEM Method
Microscope	See NIOSH 7400; as per instrument manufacturer's recommendations	See NIOSH 7400; as per instrument manufacturer's recommendations	See NIOSH 7400; as per instrument manufacturer's recommendations	Acceptable re-calibration; See NIOSH 7400	Inspect the system, correct problem, re-calibrate and/or reanalyze samples.	Non-CLP Laboratory Microscope Technician	NIOSH 7400
Microscope	See NIOSH 7402; as per instrument manufacturer's recommendations	See NIOSH 7402; as per instrument manufacturer's recommendations	See NIOSH 7402; as per instrument manufacturer's recommendations	Acceptable re-calibration; See NIOSH 7402	Inspect the system, correct problem, re-calibrate and/or reanalyze samples.	Non-CLP Laboratory Microscope Technician	NIOSH 7402
Microscope	See ASTM D5755-09 TEM Method; as per instrument manufacturer's recommendations	See ASTM D5755-09 TEM Method; as per instrument manufacturer's recommendations	See ASTM D5755-09 TEM Method; as per instrument manufacturer's recommendations	Acceptable re-calibration; See ASTM D5755-09 TEM Method	Inspect the system, correct problem, re-calibrate and/or reanalyze samples.	Non-CLP Laboratory Microscope Technician	ASTM D5755-09 TEM Method

QAPP Worksheet #26: Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): RST 3 SPM, Weston Solutions, Inc., Region II
Sample Packaging (Personnel/Organization): RST 3 SPM, Weston Solutions, Inc., Region II
Coordination of Shipment (Personnel/Organization): RST 3 SPM, Weston Solutions, Inc., Region II
Type of Shipment/Carrier: FedEx Priority Overnight
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): EPA Non-CLP Laboratory
Sample Custody and Storage (Personnel/Organization): EPA Non-CLP Laboratory
Sample Preparation (Personnel/Organization): EPA Non-CLP Laboratory
Sample Determinative Analysis (Personnel/Organization): EPA Non-CLP Laboratory
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples will be shipped or delivered within 24 hours of sample collection.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): NA
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Technicians, EPA Non-CLP Laboratories
Number of Days from Analysis: 60 days after analytical data package completed.

QAPP Worksheet #27: Sample Custody Requirements

Sample Identification Procedures: Each property will be given a unique property identification number beginning with P0001. Each sample will be labeled with the property identification number and a sample type letter code and number that depicts a specific location. Each sample will also be labeled with a Non-CLP assigned number. Depending on the type of sample, additional information such as depth, sampling round, date, etc. will be added. Examples of matrices are: S = Soil; ACM = Asbestos Containing Material; W = Wipe; A = Air Station; MV = microvacuum; PAS = Personal Air sample

Example sample locations are:

Bulk samples will be designated as: P0001-ACM001-01 (Property P0001, Bulk Sample 001, first sample collected from ACM001).
Wipe samples will be designated as: P0001-W01-01 (Property P0001, Wipe Sample 01, first sample collected from W01).
Air samples will be designated as: P0001-AS01-120313 (Property P0001, Air Station Sample 001).
Microvacuum samples will be designated as: P0001-MV01-01 (Property P0001, microvacuum Sample 01, first samples collected from MV01).
Personal air samples will be designated as: PAS01-120313 (Personnel 01, date collected 120313).

If field duplicate samples for bulk samples will be collected they will be designated with a -02 instead of a -01 at the end of the sample number.

Location of the sample collected will be recorded in the project database and site logbook. Each sample will also be labeled with a Non-CLP assigned number. Depending on the type of sample, additional information such as sampling round, date, etc. will be added.

QAPP Worksheet #27: Sample Custody Requirements (Concluded)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Each sample will be individually identified and labeled after collection, then sealed with custody seals and enclosed in a plastic cooler. The sample information will be recorded on chain-of custody (COC) forms, and the samples shipped to the appropriate laboratory via overnight delivery service or courier. COC records must be prepared in Scribe to accompany samples from the time of collection and throughout the shipping process. Each individual in possession of the samples must sign and date the sample COC Record. The COC record will be considered completed upon receipt at the laboratory. A traffic report and COC record will be maintained from the time the sample is taken to its final deposition. Every transfer of custody must be noted and signed for, and a copy of this record kept by each individual who has signed. When samples are not under direct control of the individual responsible for them, they must be stored in a locked container sealed with a custody seal. Specific information regarding custody of the samples projected to be collected on the weekend will be noted in the field logbook. The COC record should include (at minimum) the following: 1) Sample identification number; 2) Sample information; 3) Sample location; 4) Sample date; 5) Sample Time; 6) Sample Type Matrix; 7) Sample Container Type; 8) Sample Analysis Requested; 9) Name(s) and signature(s) of sampler(s); and 10) Signature(s) of any individual(s) with custody of samples.

A separate COC form must accompany each cooler for each daily shipment. The COC form must address all samples in that cooler, but not address samples in any other cooler. This practice maintains the COC for all samples in case of mis-shipment.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal): Within the laboratory, the person responsible for sample receipt must sign and date the COC form; verify that custody seals are intact on shipping containers; compare samples received against those listed on the COC form; examine all samples for possible shipping damage and improper sample preservation; note on the COC record that specific samples were damaged; notify sampling personnel as soon as possible so that appropriate samples may be regenerated; verify that sample holding times have not been exceeded; maintain laboratory COC documentation; and place the samples in the appropriate laboratory storage. At this time, no samples will be archived at the laboratory. Disposal of the samples will occur only after analyses and QA/QC checks are completed.

Note: Refer to Contract Laboratory Program Guidance for Field Samplers, EPA-540-R-09-03, January 2011:
http://www.epa.gov/superfund/programs/clp/guidance.htm#sample

QAPP Worksheet #29: Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
• Site logbooks • COC forms • Field Data Sheets • Airbills	• Samples receipt logs • Internal and external COC forms • Equipment calibration logs • Sample preparation worksheets/logs • Sample analysis worksheet/run logs • Telephone/email logs • Corrective action documentation	• Data validation reports • Field inspection checklist(s) • Laboratory audit checklist (if performed) • Review forms for electronic entry of data into database • Corrective action documentation	• Non – CLP Analytical Service Request Form

QAPP Worksheet #30: Analytical Services Table

Matrix	Analytical Group	Concentration Level	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Bulk	Asbestos (EPA 600/R-93/116 Method)	High	EPA 600/R-93/116	Two weeks verbal Three weeks written	Batta Laboratories 6 Garfield way Newark ,DE, 19713	NA
Wipe	Asbestos (ASTM D6480-05 TEM Method)	High	ASTM D6480-05 TEM Method	48 hours verbal Three days written	Batta Laboratories 6 Garfield way Newark ,DE, 19713	NA
Air	Asbestos (PCM Method)	Low	NIOSH 7400	Two weeks verbal three weeks written and 72 hours verbal Two weeks written	Batta Laboratories 6 Garfield way Newark ,DE, 19713	NA
Air	Asbestos (TEM Method)	Low	NIOSH 7402	Two weeks verbal three weeks written and 72 hours verbal Two weeks written	Batta Laboratories 6 Garfield way Newark ,DE, 19713	NA
Microvacuum	Asbestos (ASTM D5755-09 TEM Method)	High	ASTM D5755-09 TEM Method	Two weeks verbal three weeks written	Batta Laboratories 6 Garfield way Newark ,DE, 19713	NA

QAPP Worksheet #31: Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of Corrective Actions (Title and Organizational Affiliation)
Laboratory Technical Systems	Every Year	External	Regulatory Agency	Regulatory Agency	Non-CLP Laboratory	Non-CLP Laboratory	EPA or other Regulatory Agency
Peer Review	Each Deliverable	Internal	Weston Solutions, Inc.	QAO, Group Leader, and Readiness Coordinator	SPM, Weston Solutions, Inc.	SPM, Weston Solutions, Inc.	EPA OSC and/or EPA QAO

QAPP Worksheet #32: Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Project Readiness Review	Checklist or logbook entry	Carlos Huertas, Weston Solutions, Inc., RST 3	Immediately to within 24 hours of review	Checklist or logbook entry	Carlos Huertas, Weston Solutions, Inc., RST 3	Immediately to within 24 hours of review
Field Observations/Deviation from Sampling Plan	Logbook	Carlos Huertas, Weston Solutions, Inc., RST 3 and EPA OSC	Immediately to within 24 hours of review	Logbook and revision to the QAPP and/or Corrective Action Plan	Carlos Huertas, Weston Solutions, Inc., RST 3. EPA OSC and auditor	Immediately to within 24 hours of review
Laboratory Technical Systems/Performance Audit	Written Report	Non-CLP Laboratory QAO	30 days	Letter	Non-CLP Laboratory	14 days

QAPP Worksheet #33: QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Site-Specific QAPP	As performed	Prior to sampling date	Carlos Huertas, SPM, Weston Solutions, Inc., RST 3	EPA OSC
HASP	As performed	Prior to sampling date	Carlos Huertas, SPM, Weston Solutions, Inc., RST 3	EPA OSC
Trip Report (maps, photos, etc.)	As performed	Within 5 days of sample completion	Carlos Huertas, SPM, Weston Solutions, Inc., RST 3	EPA OSC and Weston Solutions, Inc., RST 3 data validator
Non-CLP laboratory data (Preliminary)	As performed	ASAP after receipt of preliminary data	Non-CLP Laboratory	SPM and EPA OSC
Non-CLP laboratory data (Validated)	As performed	* Up to 60 days after receipt of unvalidated data	Non-CLP: Data Validator, Weston Solutions, Inc., RST 3	SPM, and EPA OSC
Final Report	As specified in the site TDD	2 to 4 weeks after receipt of EPA approval of data package	Carlos Huertas, SPM, Weston Solutions, Inc., RST 3	EPA OSC

QAPP Worksheet #34: Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Site/field logbooks	Field notes will be prepared daily by the RST 3 SPM and will be complete, appropriate, legible and pertinent. Upon completion of field work, logbooks will be placed in the project files.	I	SPM, Weston Solutions, Inc.
COCs	COC forms will be reviewed against the samples packed in the specific cooler prior to shipment. The reviewer will initial the form. An original COC will be sent with the samples to the laboratory, while copies are retained for (1) the STR and (2) the project files.	I	SPM, Weston Solutions, Inc.
STRs	STRs will be prepared for each week of field sampling. Information in the STR will be reviewed against the COC forms, and potential discrepancies will be discussed with field personnel to verify locations, dates, etc.	I	SPM, Weston Solutions, Inc.
Laboratory Preliminary Data	Preliminary data – limited review for either contract compliance or technical compliance.	E	EPA non-CLP laboratory
Laboratory analytical data package	Data packages will be reviewed/verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	E	EPA non-CLP laboratory
Laboratory analytical data package	Data packages will be reviewed as to content and sample information upon receipt by Weston Solutions, Inc., RST 3	I	RST 3 Data Validator, Weston Solutions, Inc.
Final Sample Report	The project data results will be compiled in a sample report for the project. Entries will be reviewed/verified against hardcopy information.	I	SPM, Weston Solutions, Inc.

QAPP Worksheet #35: Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	SOPs	Ensure that the sampling methods/procedures outlined in QAPP were followed, and that any deviations were noted/approved.	Site Project Manager, Weston Solutions, Inc.
IIb	SOPs	Determine potential impacts from noted/approved deviations, in regard to PQOs.	Site Project Manager, Weston Solutions, Inc.
IIa	Chains of custody	Examine COC forms against QAPP and laboratory contract requirements (e.g., analytical methods, sample identification, etc.).	RST 3 Data Validator, Site Project Manager, Weston Solutions, Inc.
IIa	Laboratory data package	Examine packages against QAPP and laboratory contract requirements, and against COC forms (e.g., holding times, sample handling, analytical methods, sample identification, data qualifiers, QC samples, etc.).	RST 3 Data Validator, Site Project Manager, Weston Solutions, Inc.
IIb	Laboratory data package	Determine potential impacts from noted/approved deviations, in regard to PQOs. Examples include PQLs and QC sample limits (precision/accuracy).	RST 3 Data Validator, Site Project Manager, Weston Solutions, Inc.

QAPP Worksheet #36: Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa / IIb	Bulk	Asbestos (EPA 600/R-93/116 Method)	High	As per EPA 600/R-93/116 Method	RST 3 Data Validation Personnel, Weston Solutions, Inc.
IIa / IIb	Wipe	Asbestos (ASTM D6480-05 TEM Method)	High	As per ASTM D6480-05 TEM Method	RST 3 Data Validation Personnel, Weston Solutions, Inc.
IIa / IIb	Air	Asbestos (NIOSH 7400 PCM Method)	Low	As per NIOSH 7400 Method	RST 3 Data Validation Personnel, Weston Solutions, Inc.
IIa / IIb	Air	Asbestos (NIOSH 7402 TEM Method)	Low	As per NIOSH 7402 Method	RST 3 Data Validation Personnel, Weston Solutions, Inc.
IIa / IIb	Microvacuum	Asbestos (ASTM D5755-09 TEM Method)	High	As per ASTM D5755-09 Method	RST 3 Data Validation Personnel, Weston Solutions, Inc.

QAPP Worksheet #37: Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: Data, whether generated in the field or by the laboratory, are tabulated and reviewed for Precision, Accuracy, Representativeness, Completeness, and Comparability (PARCCS) by the SPM for field data or the data validator for laboratory data. The review of the PARCC Data Quality Indicators (DQI) will compare with the DQO detailed in the site-specific QAPP, the analytical methods used and impact of any qualitative and quantitative trends will be examined to determine if bias exists. A hard copy of field data is maintained in a designated field or site logbook. Laboratory data packages are validated, and final data reports are generated. All documents and logbooks are assigned unique and specific control numbers to allow tracking and management.

Questions about non-CLP data, as observed during the data review process, are resolved by contacting the respective site personnel and laboratories as appropriate for resolution. All communications are documented in the data validation record with comments as to the resolution to the observed deficiencies.

Where applicable, the following documents will be followed to evaluate data for fitness in decision making: EPA QA/G-4, *Guidance on Systematic Planning using the Data Quality Objectives Process,* EPA/240/B-06/001, February 2006, and EPA QA/G-9R, *Guidance for Data Quality Assessment*, A reviewer's Guide EPA/240/B-06/002, February 2006.

Describe the evaluative procedures used to assess overall measurement error associated with the project: As delineated in the ***Uniform Federal Policy for Implementing Environmental Quality Systems: Evaluating, Assessing and Documenting Environmental Data Collection and Use Programs Part 1: UFP-QAPP (EPA-505-B-04-900A, March 2005); Part 2A: UFP-QAPP Workbook (EPA-505-B-04-900C, March 2005); Part 2B: Quality Assurance/Quality Control Compendium: Non-Time Critical QA/QC Activities (EPA-505-B-04-900B, March 2005);*** "Graded Approach" will be implemented for data collection activities where specific decisions cannot be identified, since this guidance indicates that the formal DQO process is not necessary.

QAPP Worksheet #37: Usability Assessment (Concluded)

Sampling will be conducted by RST 3 to identify/confirm the presence of asbestos. The data will be used by EPA to determine if classrooms were cleaned properly and to identify construction materials containing asbestos. If air samples collected at the facility are above 0.1 f/cc, PPE Level D will be upgraded to Level C.

Identify the personnel responsible for performing the usability assessment: Site Project Management Team, and EPA, Region II OSC

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: A copy of the most current approved QAPP, including any graphs, maps and text reports developed will be provided to all personnel identified on the distribution list.

ATTACHMENT A

FIGURE 1 – SITE LOCATION MAP

Legend

Site Location

0 0.05 0.1 0.2 0.3 0.4 Miles

N

Weston Solutions, Inc.
East Division

In Association With
H & S Environmental, Inc.,
Scientific and Environmental Associates, Inc.
and Avatar Environmental, LLC.

Figure 1
Site Location Map

Puerto Rico Olefins Asbestos
Penuelas, Puerto Rico

U.S. ENVIRONMENTAL PROTECTION AGENCY
REMOVAL SUPPORT TEAM 2
CONTRACT # EP-W-06-072

GIS ANALYST:	T. BENTON
EPA OSC:	A. RODRIGUEZ
RST SPM:	C. HUERTAS
FILENAME:	SITEMAP.MXD

DATE MODIFIED: 12/26/2013

ATTACHMENT B

SAMPLING SOPS

GENERAL FIELD SAMPLING GUIDELINES

SOP#: 2001
DATE: 08/11/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide general field sampling guidelines that will assist REAC personnel in choosing sampling strategies, location, and frequency for proper assessment of site characteristics. This SOP is applicable to all field activities that involve sampling.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Sampling is the selection of a representative portion of a larger population, universe, or body. Through examination of a sample, the characteristics of the larger body from which the sample was drawn can be inferred. In this manner, sampling can be a valuable tool for determining the presence, type, and extent of contamination by hazardous substances in the environment.

The primary objective of all sampling activities is to characterize a hazardous waste site accurately so that its impact on human health and the environment can be properly evaluated. It is only through sampling and analysis that site hazards can be measured and the job of cleanup and restoration can be accomplished effectively with minimal risk. The sampling itself must be conducted so that every sample collected retains its original physical form and chemical composition. In this way, sample integrity is insured, quality assurance standards are maintained, and the sample can accurately represent the larger body of material under investigation.

The extent to which valid inferences can be drawn from a sample depends on the degree to which the sampling effort conforms to the project's objectives. For example, as few as one sample may produce adequate, technically valid data to address the project's objectives. Meeting the project's objectives requires thorough planning of sampling activities, and implementation of the most appropriate sampling and analytical procedures. These issues will be discussed in this procedure.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The amount of sample to be collected, and the proper sample container type (i.e., glass, plastic), chemical preservation, and storage requirements are dependent on the matrix being sampled and the parameter(s) of interest. Sample preservation, containers, handling, and storage for air and waste samples are discussed in the specific SOPs for air and waste sampling techniques.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The nature of the object or materials being sampled may be a potential problem to the sampler. If a material is homogeneous, it will generally have a uniform composition throughout. In this case, any sample increment can be considered representative of the material. On the other hand, heterogeneous samples present problems to the sampler because of changes in the material over distance, both laterally and vertically.

Samples of hazardous materials may pose a safety threat to both field and laboratory personnel. Proper health and safety precautions should be implemented when handling this type of sample.

Environmental conditions, weather conditions, or non-target chemicals may cause problems and/or interferences when performing sampling activities or when sampling for a specific parameter. Refer to the specific SOPs for sampling techniques.

5.0 EQUIPMENT/APPARATUS

The equipment/apparatus required to collect samples must be determined on a site specific basis. Due to the wide variety of sampling equipment available, refer to the specific SOPs for sampling techniques which include lists of the equipment/apparatus required for sampling.

6.0 REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed. Decontamination solutions are specified in ERT SOP #2006, Sampling Equipment Decontamination.

7.0 PROCEDURE

7.1 Types of Samples

In relation to the media to be sampled, two basic types of samples can be considered: the environmental sample and the hazardous sample.

Environmental samples are those collected from streams, ponds, lakes, wells, and are off-site samples that are not expected to be contaminated with hazardous materials. They usually do not require the special handling procedures typically used for concentrated wastes. However, in certain instances, environmental samples can contain elevated concentrations of pollutants and in such cases would have to be handled as hazardous samples.

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, or areas previously identified as contaminated, and require special handling procedures because of their potential toxicity or hazard. These samples can be further subdivided based on their degree of hazard; however, care should be taken when handling and shipping any wastes believed to be concentrated regardless of the degree.

The importance of making the distinction between environmental and hazardous samples is two-fold:

(1) Personnel safety requirements: Any sample thought to contain enough hazardous materials to pose a safety threat should be designated as hazardous and handled in a manner which ensures the safety of both field and laboratory personnel.

(2) Transportation requirements: Hazardous samples must be packaged, labeled, and shipped according to the International Air Transport Association (IATA) Dangerous Goods Regulations or Department of Transportation (DOT) regulations and U.S. EPA guidelines.

7.2 Sample Collection Techniques

In general, two basic types of sample collection techniques are recognized, both of which can be used for either environmental or hazardous samples.

Grab Samples

A grab sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected all at once at one particular point in the sample medium. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

Composite Samples

Composites are nondiscrete samples composed of more than one specific aliquot collected at various sampling locations and/or different points in time. Analysis of this type of sample produces an average value and can in certain instances be used as an alternative to analyzing a number of individual grab samples and calculating an average value. It should be noted, however, that compositing can mask problems by diluting isolated concentrations of some hazardous compounds below detection limits.

Compositing is often used for environmental samples and may be used for hazardous samples under certain conditions. For example, compositing of hazardous waste is often performed after compatibility tests have

been completed to determine an average value over a number of different locations (group of drums). This procedure generates data that can be useful by providing an average concentration within a number of units, can serve to keep analytical costs down, and can provide information useful to transporters and waste disposal operations.

For sampling situations involving hazardous wastes, grab sampling techniques are generally preferred because grab sampling minimizes the amount of time sampling personnel must be in contact with the wastes, reduces risks associated with compositing unknowns, and eliminates chemical changes that might occur due to compositing.

7.3 Types of Sampling Strategies

The number of samples that should be collected and analyzed depends on the objective of the investigation. There are three basic sampling strategies: random, systematic, and judgmental sampling.

Random sampling involves collection of samples in a nonsystematic fashion from the entire site or a specific portion of a site. Systematic sampling involves collection of samples based on a grid or a pattern which has been previously established. When judgmental sampling is performed, samples are collected only from the portion(s) of the site most likely to be contaminated. Often, a combination of these strategies is the best approach depending on the type of the suspected/known contamination, the uniformity and size of the site, the level/type of information desired, etc.

7.4 QA Work Plans (QAWP)

A QAWP is required when it becomes evident that a field investigation is necessary. It should be initiated in conjunction with, or immediately following, notification of the field investigation. This plan should be clear and concise and should detail the following basic components, with regard to sampling activities:

- Objective and purpose of the investigation.
- Basis upon which data will be evaluated.
- Information known about the site including location, type and size of the facility, and length of operations/abandonment.
- Type and volume of contaminated material, contaminants of concern (including concentration), and basis of the information/data.
- Technical approach including media/matrix to be sampled, sampling equipment to be used, sample equipment decontamination (if necessary), sampling design and rationale, and SOPs or description of the procedure to be implemented.
- Project management and reporting, schedule, project organization and responsibilities, manpower and cost projections, and required deliverables.
- QA objectives and protocols including tables summarizing field sampling and QA/QC analysis and objectives.

Note that this list of QAWP components is not all-inclusive and that additional elements may be added or altered depending on the specific requirements of the field investigation. It should also be recognized that although a detailed QAWP is quite important, it may be impractical in some instances. Emergency responses and accidental spills are prime examples of such instances where time might prohibit the development of site-specific QAWPs prior to field activities. In such cases, investigators would have to rely on general guidelines and personal judgment, and the sampling or response plans might simply be a strategy based on preliminary information and finalized on site. In any event, a plan of action should be developed, no matter how concise or informal, to aid investigators in maintaining a logical and consistent order to the implementation of their task.

7.5 Legal Implications

The data derived from sampling activities are often introduced as critical evidence during litigation of a hazardous waste site cleanup. Legal issues in which sampling data are important may include cleanup cost recovery, identification of pollution sources and responsible parties, and technical validation of remedial design methodologies. Because of the potential for involvement in legal actions, strict adherence to technical and administrative SOPs is essential during both the development and implementation of sampling activities.

Technically valid sampling begins with thorough planning and continues through the sample collection and analytical procedures. Administrative requirements involve thorough, accurate

documentation of all sampling activities. Documentation requirements include maintenance of a chain of custody, as well as accurate records of field activities and analytical instructions. Failure to observe these procedures fully and consistently may result in data that are questionable, invalid and non-defensible in court, and the consequent loss of enforcement proceedings.

8.0 CALCULATIONS

Refer to the specific SOPs for any calculations which are associated with sampling techniques.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Refer to the specific SOPs for the type and frequency of QA/QC samples to be analyzed, the acceptance criteria for the QA/QC samples, and any other QA/QC activities which are associated with sampling techniques.

10.0 DATA VALIDATION

Refer to the specific SOPs for data validation activities that are associated with sampling techniques.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures.

GENERAL AIR SAMPLING GUIDELINES

SOP#: 2008
DATE: 11/16/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) provides guidance in developing and implementing sampling plans to assess the impact of hazardous waste sites on ambient air. It presents the United States Environmental Protection Agency/Environmental Response Team's (U.S. EPA/ERT's) approach to air sampling and monitoring and identifies equipment requirements. It is not within the scope of this SOP to provide a generic air sampling plan. Experience, objectives, site characteristics, and chemical characteristics will dictate sampling strategy. This SOP does not address indoor air sampling.

Two basic approaches can be used to assess ambient air (also referred to as air pathway assessments): modeling and measurements. The modeling approach initially estimates or measures the overall site emission rate(s) and pattern(s). These data are input into an appropriate air dispersion model, which predicts either the maximum or average air concentrations at selected locations or distances during the time period of concern. This overall modeling strategy is presented in the first three volumes of the Air Superfund National Technical Guidance Series on Air Pathway Assessments[1,2,3]. Specific applications of this strategy are presented in several additional Air Superfund Technical Guidance documents[4].

The measurement approach involves actually measuring the air impact at selected locations during specific time periods. These measurements can be used to document actual air impacts during specific time intervals (i.e., during cleanup operations) or to extrapolate the probable "worst case" concentrations at that and similar locations over a longer time period than was sampled.

This SOP addresses issues associated with this second assessment strategy. This SOP also discusses the U.S. EPA/ERT's monitoring instruments, air sampling kits, and approach to air sampling and monitoring at hazardous waste sites.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, depending on site conditions, equipment limitations, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Air monitoring is defined as the use of direct-reading instruments and other screening or monitoring equipment and techniques that provide instantaneous (real-time) data on the levels of airborne contaminants. The U.S. EPA/ERT maintains numerous monitors for real-time measurements. Examples of air monitoring equipment are hand-held photoionization detectors (PID), flame ionization detectors (FID), oxygen/combustible gas detectors, and remote optical sensors.

Air sampling is defined as those sampling and analytical techniques that require either off- or on-site laboratory analysis and therefore do not provide immediate results. Typically, air sampling occurs after use of real-time air monitoring equipment has narrowed the number of possible contaminants and has provided some qualitative measurement of contaminant concentration. Air sampling techniques are used to more accurately detect, identify and quantify specific chemical compounds relative to the majority of air monitoring technologies.

In the Superfund Removal Program, On-Scene Coordinators (OSCs) may request the U.S. EPA/ERT to conduct air monitoring and sampling during the

following situations: emergency responses, site assessments, and removal activities. Each of these activities has a related air monitoring/sampling objective that is used to determine the potential hazards to workers and/or the community.

C Emergency Response

Emergency responses are immediate responses to a release or threatened release of hazardous substances presenting an imminent danger to public health, welfare, or the environment (i.e., chemical spills, fires, or chemical process failures resulting in a controlled release of hazardous substances). Generally these situations require rapid on-site investigation and response. A major part of this investigation consists of assessing the air impact of these releases.

C Removal Site Assessment

Removal site assessments (referred to as site assessments) are defined as any of several activities undertaken to determine the extent of contamination at a site and which help to formulate the appropriate response to a release or threatened release of hazardous substances. These activities may include a site inspection, multimedia sampling, and other data collection.

C Removal Actions

Removal actions clean up or remove hazardous substances released into the environment. Removal actions include any activity conducted to abate, prevent, minimize, stabilize, or eliminate a threat to public health or welfare, or to the environment.

Personal risk from airborne contaminants can be determined by comparing the results of on-site monitoring and sampling to health-based action levels such as the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) and the Occupational Safety and Health Administration (OSHA) Permissible Exposure Limits (PELs). Residential risk can be determined by comparing the results of off-site monitoring or sampling to health-based action levels such as those developed by the Agency for Toxic Substance and Disease Registry (ATSDR).

The extent to which valid inferences can be drawn from air monitoring/sampling depends on the degree to which the monitoring/sampling effort conforms to the objectives of the event. Meeting the project's objectives requires thorough planning of the monitoring/sampling activities, and implementation of the most appropriate monitoring/sampling and analytical procedures. These issues will be discussed in this SOP.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Preservation, containers, handling and storage for air samples are discussed in the specific SOPs for the technique selected. In addition, the analytical method (i.e., U.S. EPA, National Institute for Occupational Safety and Health [NIOSH], and OSHA Methods) may be consulted for storage temperature, holding times and packaging requirements. After sample collection, the sampling media (i.e., cassettes or tubes) are immediately sealed. The samples are then placed into suitable containers (i.e., whirl bags, resealable bagsor culture tubes) which are then placed into a shipping container.

Use bubble wrap or styrofoam peanuts when packing air samples for shipment. DO NOT USE VERMICULITE.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Upwind sources can contribute to sample concentration. Natural sources, such as biological waste, can produce hydrogen sulfide and methane which may contribute to the overall contaminant level. Extraneous anthropogenic contaminants (i.e., burning of fossil fuels; emissions from vehicular traffic, especially diesel; volatile compounds from petrochemical facilities; and effluvium from smoke stacks) may also contribute. Air sampling stations should be strategically placed to identify contributing sources.

Photoreactivity or reaction of the parameters of concern may occur with nonrelated compounds [i.e., nitrogen compounds and polyaromatic hydrocarbons

(PAHs)]. Some sorbent media/samples should not be exposed to light during or after sampling due to photochemical effects (i.e., PAHs).

Various environmental factors, including humidity, temperature and pressure, also impact the air sampling methodology, collection efficiency and detection limit. Since the determination of air contaminants is specifically dependent on the collection parameters and efficiencies, the collection procedure is an integral part of the analytical method.

Detection limits depend on the contaminants being investigated and the particular site situation. It is important to know why the data are needed and how the data will be used. Care should be taken to ensure the detection limits are adequate for the intended use of the final results.

Some equipment may be sensitive to humidity and temperature extremes.

5.0 EQUIPMENT/APPARATUS

5.1 Direct Reading Instruments (Air Monitoring Instruments)

There are two general types of direct reading instruments: portable screening devices and specialized analytical instruments. Generally all these techniques involve acquiring, for a specific location or area, continuous or sequential direct air concentrations in either a real-time or semi-real-time mode. None of these instruments acquires true time-weighted average concentrations. In addition, these instruments are not capable of acquiring simultaneous concentration readings at multiple locations, although several are able to sequentially analyze samples taken remotely from different locations. The document, "Guide to Portable Instruments for Assessing Airborne Pollutants Arising from Hazardous Waste Sites[5]," provides additional information about air sampling and monitoring. The hazard levels for airborne contaminants vary. See the ACGIH TLVs and the OSHA PELs for safe working levels. Common screening devices and analytical instruments are described in Appendix A.

5.2 Air Sampling Equipment and Media/Devices

The U.S. EPA/ERT uses the following analytical methods for sampling: *NIOSH Manual of Analytical Methods*[6], *American Society for Testing and Materials (ASTM) Methods*[7], *U.S. EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*[8,9], and *OSHA Methods*[10]. Additional air sampling references include *Industrial Hygiene and Toxicology* (3rd Ed.)[11] and *Air Sampling Instruments for Evaluation of Atmospheric Contaminants*[12]. These methods typically specify equipment requirements for sampling. Since air sampling is such a diverse technology, no single method or reference is best for all applications. Common sampling equipment and media/devices are described in Appendix B.

5.3 Tools/Material and Equipment List

In addition to equipment and materials identified in Appendices A and B, the following equipment and materials may be required to conduct air sampling and monitoring at hazardous waste sites:

- Camera
- Site logbook
- Clipboard
- Chain of custody records
- Custody seals
- Air sampling worksheets
- Sample labels
- Small screwdriver set
- Aluminum foil
- Extension cords
- Glass cracker
- Multiple plug outlet
- Whirl bags or culture tubes
- Teflon tape
- Calibration devices
- Tygon and/or Teflon^{R} tubing
- Surgical gloves
- Lint-free gloves
- Ice
- Sample container

Use the following additional equipment when decontaminating glassware on site:

- Protective equipment (i.e., gloves, splash goggles, etc.)
- Appropriate solvent(s)
- Spray bottles
- Liquinox (soap)
- Paper towels

- C Distilled/deionized water
- C Five-gallon buckets
- C Scrub brushes and bottle brushes

6.0 REAGENTS

Impinger sampling involves using reagents contained in a glass vial to absorb contaminants of concern (for example, NIOSH Method 3500 for formaldehyde uses 1% sodium bisulfite solution). Impinger solutions vary and are method-dependent.

Reagents such as acetone and hexane are required to decontaminate glassware and some air sampling equipment. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP.

7.0 PROCEDURES

7.1 Air Monitoring Design

7.1.1 Initial Surveys

In general, the initial survey is considered to be a relatively rapid screening process for collecting preliminary data at hazardous waste sites. However, initial surveys may require many hours to complete and may consist of more than one entry.

Some information is generally known about the site; therefore, real-time instrumentation for specific compounds (i.e., detector tubes and electrochemical sensors) can be used to identify hot spots. Sufficient data should be obtained with real-time instruments during the initial entry to screen the site for various contaminants. When warranted, intrinsically safe or explosion-proof instruments should be used. An organic vapor analyzer (OVA) is typically used during this survey. These gross measurements may be used on a preliminary basis to (1) determine levels of personal protection, (2) establish site work zones, and (3) map candidate areas for more thorough qualitative and quantitative studies involving air sampling.

In some situations, the information obtained may be sufficient to preclude additional monitoring. Materials detected during the initial survey may call for a more comprehensive evaluation of hazards and analyses for specific compounds. Since site activities and weather conditions change, a continuous program to monitor the ambient atmosphere must be established.

7.1.2 Off-Site Monitoring

Typically, perimeter monitoring with the same instruments employed for on-site monitoring is utilized to determine site boundaries. Because air is a dynamic matrix, physical boundaries like property lines and fences do not necessarily delineate the site boundary or area influenced by a release. Whenever possible, atmospheric hazards in the areas adjacent to the on-site zone should be monitored with direct-reading instruments. Monitoring at the fenceline or at varying locations off site provides useful information regarding pollutant migration. Three to four locations downwind of the source (i.e., plume) at breathing-zone height, provide a basic fingerprint of the plume. Negative instrument readings off site should not be interpreted as the complete absence of airborne toxic substances; rather, they should be considered another piece of information to assist in the preliminary evaluation. The interpretation of negative readings is instrument-dependent. The lack of instrument readings off site should not be interpreted as the complete absence of all airborne toxic substances; rather, it is possible that the particular compound or class of compounds to which the monitoring instrument responds is not present or that the concentration of the compound(s) is below the instrument's detection limit.

7.2 Air Sampling Design

7.2.1 Sampling Plan Design

The goal of air sampling is to accurately assess the impact of a contaminant source(s) on ambient air quality. This impact is expressed in terms of overall average and/or maximum air concentrations for the time period of concern and may be affected by the transport and release of pollutants from both on- and off-site sources. The location of these sources must be taken into account as they impact the selection of sampling locations. Unlike soil and groundwater concentrations, air concentrations at points of interest can easily vary by orders of magnitude over the period of concern. This variability plays a major role in designing an air sampling plan.

Downwind air concentration is determined by the amount of material being released from the site into the air (the emission rate) and by the degree that the contamination is diluted as it is transported. Local

meteorology and topography govern downwind dilution. Contaminant emission rates can also be heavily influenced by on-site meteorology and on-site activities. All of these concerns must be incorporated into an air sampling plan.

A sampling strategy can be simple or complex, depending on the sampling program objectives. Programs involving characterization of the pollutant contribution from a single point source tend to be simple, whereas sampling programs investigating fate and transport characteristics of components from diverse sources require a more complex sampling strategy. In addition, resource constraints may affect the complexity of the sampling design.

An optimal sampling strategy accounts for the following site parameters:

- Location of stationary as well as mobile sources
- Analytes of concern
- Analytical detection limit to be achieved
- Rate of release and transport of pollutants from sources
- Availability of space and utilities for operating sampling equipment
- Meteorological monitoring data
- Meteorological conditions in which sampling is to be conducted

The sampling strategy typically requires that the concentration of contaminants at the source or area of concern as well as background contributions be quantified. It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks, as well as various other types of QA/QC samples, can be utilized to determine other sources. The impact of extraneous sources on sampling results can frequently be accounted for by placing samplers upwind, downwind and crosswind from the subject source. The analytical data from these different sampling locations may be compared to determine statistical differences.

7.2.2 Sampling Objectives

The objectives of the sampling must be determined prior to developing the sampling plan. Does the sampling plan verify adequate levels of protection for on-site personnel, or address potential off-site impacts associated with the site or with site activities? In addition, the assumptions associated with the sampling program must be defined. These assumptions include whether the sampling is to take place under "typical," "worst case," or "one-time" conditions. If the conditions present at the time of sampling are different from those assumed during the development of the sampling plan, then quality of the data collected may be affected. The following definitions have been established:

- Typical: routine daily sampling or routine scheduled sampling at pre-established locations.

- Worst case: sampling conducted under the worst meteorological and/or site conditions which would result in elevated ambient concentrations.

- One-time: only one chance is given to collect a sample without regard to time or conditions.

Qualitative data acquired under these conditions are usually applicable only to the time period during which the data were collected and may not provide accurate information to be used in estimating the magnitude of an air impact during other periods or over a long time interval.

The sampling objectives also dictate the detection limits. Sampling methods for airborne contaminants will depend upon the nature and state (solid, liquid or gas) of the contaminant. Gases and vapors may be collected in aqueous media or adsorbents, in molecular sieves, or in suitable containers. Particulates are collected by filters or impactors. The volume of sample to be collected is dependent upon an estimate of the contaminant concentration in the air, the sensitivity of the analytical method, and the standard or desired detection limit. A sufficient amount of sample must be collected to achieve the desired detection limit without interference from other contaminants. In addition, the selected method must be able to detect the target compound(s).

7.2.3 Location and Number of Individual Sampling Points

Choose the number and location of sampling points according to the variability, or sensitivity, of the

sampling and analytical methods being utilized, the variability of contaminant concentration over time at the site, the level of precision required and costlimitations. In addition, determine the number of locations and placement of samplers by considering the nature of the response, local terrain, meteorological conditions, location of the site (with respect to other conflicting background sources), size of the site, and the number, size, and relative proximity of separate on-site emission sources and upwind sources. The following are several considerations for sampler placement:

- Location of potential on-site emission sources, as identified from the review of site background information or from preliminary on-site inspections.

- Location of potential off-site emission sources upwind of the sampling location(s). Review local wind patterns to determine the location of off-site sources relative to wind direction.

- Topographic features that affect the dispersion and transport of airborne toxic constituents.

Avoid natural obstructions when choosing air sampling station locations, and account for channelization around those obstructions.

- Large water bodies, which affect atmospheric stability and the dispersion of air contaminants.

- Roadways (dirt or paved), which may generate dust that could mask site contaminants.

- Vegetation, such as trees and shrubs, which stabilizes soil and retards subsurface contaminants from becoming airborne. It also affects air flow and scrubs some contaminants from the air. Sometimes thick vegetation can make an otherwise ideal air monitoring location inaccessible.

Consider the duration of sampling activities when choosing the location and number of samples to be collected. For example, if the sampling period is limited to a few hours, one or two upwind and several downwind samples would typically be adequate, especially around major emission sources.

A short-term monitoring program ranges from several days to a few weeks and generally includes gathering data for site assessments, removal actions, and source determination data (for further modeling). Activities involved in a short-term sampling strategy must make the most of the limited possibilities for data collection. Consider moving upwind/downwind locations daily based on National Oceanic and Atmospheric Administration (NOAA) weather forecasts. Weather monitoring becomes critical where complex terrain and local meteorological effects frequently change wind direction. Often, a number of alternatives can fulfill the same objective.

Prevailing winds running the length of a valley usually require a minimum number of sampler locations; however, a complex valley may require more sampler locations to account for the wide variety of winds. Ocean/lake effects may require a radical plan to collect enough samples to reach a low detection limit. Two sets of samplers may be placed next to each other: one set would be activated during the sea breeze while the other set is turned off, and vice versa when there is no sea breeze. After the sampling event, the respective upwind and downwind samples would be combined. Another alternative for sampling near a large body of water may be to use automatic, wind-vector-operated samplers, which turn the sampler on only when the wind comes from a specified vector. At sites located on hillsides, wind will move down a valley and produce an upward fetch at the same time. Sampling locations may have to ring the site to measure the wind's impact.

Off-site sources may affect on-site monitoring. In this case, on-site meteorological data, concurrent with sampling data, is essential to interpreting the acquired data. Also, additional upwind sampling sites may be needed to fully characterize ambient background contaminant levels. Multiple off-site sources may require several monitoring locations, but if the sources are at a sufficient distance, only one monitoring location is needed.

Topography and weather are not the only factors in sampler location; the sampling sites must be secure from vandals and mishap. Secure all sampling locations to maintain chain of custody, and to prevent tampering with samples or loss of sampling units. High-volume sampling methods often require the use of 110 VAC electric power. When portable

generators are used, the power quality may affect sampler operation. Also, be aware that the generators themselves could be a potential pollution source if their placement is not carefully considered.

Air quality dispersion models can be used to place samplers. The models incorporate source information, surrounding topography, and meteorological data to predict the general distance and directions of maximum ambient concentrations. Modeling results should be used to select sampling locations in areas of maximum pollutant concentrations.

7.2.4 Time, Duration and Frequency of Sampling Events

After choosing appropriate sampling or monitoring locations, determine the sampling frequency and the number of samples to be collected. The time of day, duration and frequency of sampling events is governed by:

- The effects of site activities and meteorology on emission rates
- The diurnal effect of the meteorology on downwind dispersion
- The time period(s) of concern as defined by the objective
- The variability in the impact from other non-site-related sources
- If defined, the degree of confidence needed for either the mean or maximum downwind concentrations observed
- The precision requirements for single measurements
- Cost and other logistical considerations

The duration of the removal action and the number of hours per day that site work is conducted determine the time, duration, and frequency of samples. Short-term sampling programs may require daily sampling, while long-term programs may require 24-hour sampling every sixth or twelfth day. If the site will be undergoing removal activities 24 hours a day, continuous air sampling may be warranted. However, if the site activities will be conducted for only eight hours a day, and there are no emissions likely to occur during the remaining 16 hours, then sampling would be appropriate prior to the start of daily activities, would continue during operations, and end at the conclusion of the daily activities. An off-peak sample collection can ensure that emissions are not persisting after the conclusion of daily cleanup activities. For some sites, emissions are still a factor several hours after daily site activities have been completed. Because of the typically decreased downwind dispersion in the evening, higher downwind concentrations than were present during daytime site activities may be detected. For sites where this is possible, the sampling duration needs to be lengthened accordingly.

Sampling duration and flow rate dictate the volume of air collected, and to a major degree, the detection limit. The analytical method selected will provide a reference to flow rate and volume. Flow rates are limited to the capacity of the pumps being employed and the contact time required by the collection media.

The duration or period of air sampling is commonly divided into two categories (1) samples collected over a brief time period are referred to as "instantaneous" or "grab" samples and are usually collected in less than five minutes and (2) average or integrated samples are collected over a significantly longer period of time. Integrated samples provide an average concentration over the entire sampling period. Integrated samples are not suited to determining cyclical releases of contaminants because periodic or cyclical events are averaged out by the proportionally long sampling duration.

Air quality dispersion models can predict the maximum air contaminant concentration expected from a source. The meteorological and site conditions expected to cause the highest concentration are known as worst-case conditions and can be identified by analyzing the modeling results. Depending upon the objective, one may sample when the model predicts worst-case conditions will exist.

7.2.5 Meteorological and Physical/Chemical Considerations

A meteorological monitoring program is an integral part of site monitoring activities. Meteorological data, which define local terrain impacts on air flow paths, are needed to interpret air concentration data. Meteorological data may be available from an existing station located near the site (i.e., at a local airport), otherwise a station should be set up at the site. This data will document the degree that samples actually were downwind and verify whether other worst-case assumptions were met. Meteorological parameters to

be monitored are, at a minimum, wind speed, wind direction, and sigma theta (which is the horizontal wind direction standard deviation and an indicator of atmospheric stability). The remaining parameters primarily affect the amount of a contaminant available in the air.

C Wind Speed

When the contaminant of concern is a particulate, wind speed is critical in determining whether the particulate will become airborne, the quantity of the particulate that becomes airborne, and the distance the particulate will travel from the source. Wind speed also contributes to the volatilization of contaminants from liquid sources.

C Wind Direction

Wind direction highly influences the path of airborne contaminants. In addition, variations in wind direction increase the dispersion of pollutants from a given source.

C Atmospheric Stability

Atmospheric stability refers to the degree to which the atmosphere tends to dampen vertical and horizontal motion. Stable atmospheric conditions (i.e., evenings) result in low dispersion, and unstable atmospheric conditions (i.e., hot sunny days) result in higher dispersion.

C Temperature

Higher temperatures increase the rate of volatilization of organic and some inorganic compounds and affect the initial rise of gaseous or vapor contaminants. Therefore, worst-case emission of volatiles and semivolatiles occurs at the hottest time of day, or on the hottest day.

C Humidity

High humidity affects water-soluble chemicals and particulates. Humid conditions may dictate the sampling media used to collect the air sample, or limit the volume of air sampled and thereby increase the detection limit.

C Atmospheric Pressure

Migration of landfill gases through the landfill surface and through surrounding soils are governed by changes in atmospheric pressure. Atmospheric pressure will influence upward migration of gaseous contaminants from shallow aquifers into the basements of overlying structures.

In many cases, the transport and dispersion of air pollutants is complicated by local meteorology. Normal diurnal variations (i.e., temperature inversions) affect dispersion of airborne contaminants. Terrain features can enhance or create air inversions and can also influence the path and speed of air flow, complicating transport and dispersion patterns.

The chemical characteristics of a contaminant (i.e., molecular weight, physical state, vapor pressure, aerodynamic size, temperature, reactive compounds, and photodegradation) affects its behavior and can influence the method used to sample and analyze it.

8.0 CALCULATIONS

Volume is obtained by multiplying the sample time in minutes by the flow rate. Sample volume should be indicated on the chain of custody record. Adjustments for temperature and pressure differences may be required.

Results are usually provided in parts per million (ppm), parts per billion (ppb), milligrams per cubic meter (mg/m^3) or micrograms per cubic meter ($\mu g/m^3$).

Refer to the analytical method or regulatory guidelines for other applicable calculations.

9.0 QUALITYASSURANCE/ QUALITY CONTROL

The manufacturer's instructions should be reviewed prior to instrument use. Instruments must be utilized in accordance with manufacturer's instructions. Equipment checkout and calibration activities must

occur prior to and after monitoring and sampling and must be documented.

9.1 QA/QC Samples

QA/QC samples provide information on the variability and usability of environmental sample results. Various QA/QC samples may be collected to detect error. QA/QC samples are submitted with the field samples for analysis to aid in identifying the origin of analytical discrepancies; then a determination can be made as to how the analytical results should be used. Collocated samples, background samples, field blanks, and lot blanks are the most commonly collected QA/QC field samples. Performance evaluation (PE) samples and matrix spikes provide additional measures of data QA/QC control. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet site-specific QA or data quality objectives.

9.2 Sample Documentation

All sample and monitoring activities should be documented legibly, in ink. Any corrections or revisions should be made by lining through the incorrect entry and by initialing the error. All samples must be recorded on an Air Sampling Worksheet. A chain of custody record must be maintained from the time a sample is taken to the final deposition of the sample. Custody seals demonstrate that a sample container has not been opened or tampered with during transport or storage of samples.

10.0 DATA VALIDATION

Results for QA/QC samples should be evaluated for contamination. This information should be utilized to qualify the environmental sample results accordingly with data quality objectives.

11.0 HEALTH AND SAFETY

Personal protection equipment (PPE) requirements identified in federal and/or state regulations and 29 Code of Federal Regulations (CFR) 1910.120 for hazardous waste site work must be followed.

The majority of physical precautions involved in air sampling are related to the contaminant sampled. Attention should be given when sampling in potentially explosive, flammable or acidic atmospheres. On rare occasions, the collection media may be hazardous; for example, in the instance where an acidic or basic solution is utilized in an impinger.

When working with potentially hazardous materials, follow U.S. EPA, OSHA and corporate health and safety procedures.

12.0 REFERENCES

(1) U.S. EPA. *Air Superfund National Technical Guidance Series. Volume I. Application of Air Pathway Analyses for Superfund Activities*. EPA/450/1-89/001.

(2) U.S. EPA. *Air Superfund National Technical Guidance Series. Volume II. Estimation of Baseline Air Emissions at Superfund Sites*. EPA/450/1-89/002.

(3) U.S. EPA. *Air Superfund National Technical Guidance Series. Volume III. Estimations of Air Emissions from Cleanup Activities at Superfund Sites*. EPA/450/1-89/003.

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APPENDIX A

Portable Screening Devices and Specialized Analytical Instruments

PORTABLE SCREENING DEVICES

Where possible, a datalogger should be used to minimize the length of time required for site personnel to be in a potentially contaminated area. Datalogger cable is available from manufacturers for linear output instruments and some nonlinear output instruments. U.S. EPA ERT/REAC has output cables for organic vapor analyzers (i.e., HNU and OVA), toxic gas analyzers (i.e., monitox) and real-time aerosol monitors (i.e., RAM and miniram).

C Total Hydrocarbon Analyzers

Total hydrocarbon analyzers used to detect a variety of volatile organic compounds (VOCs) at hazardous waste sites principally employ either a photoionization detector (PID) or a flame ionization detector (FID). Compounds are ionized by a flame or an ultraviolet lamp. PIDs depend on the ionization potential of the compounds. PIDs are sensitive to aromatic and olefinic (unsaturated) compounds such as benzene, toluene, styrene, xylenes, and acetylene. Greater selectivity is possible by using low-voltage lamps. The ionization potential of individual compounds can be found in the NIOSH Pocket Guide to Chemical Hazards. These instruments are not compound-specific and are typically used as screening instruments. FIDs are sensitive to volatile organic vapor compounds such as methane, propanol, benzene and toluene. They respond poorly to organic compounds lacking hydrocarbon characteristics.

C Oxygen and Combustible Gas Indicators

Combustible Gas Indicators (CGIs) provide efficient and reliable methods to test for potentially explosive atmospheres. CGI meters measure the concentration of a flammable vapor or gas in air and present these measurements as a percentage of the lower explosive limit (LEL).

The measurements are temperature-dependent. The property of the calibration gas determines sensitivity. LELs for individual compounds can be found in the NIOSH Pocket Guide to Chemical Hazards. If readings approach or exceed 10% of the LEL, extreme caution should be exercised in continuing the investigation. If readings approach or exceed 25% LEL, personnel should be withdrawn immediately.

CGIs typically house an electrochemical sensor to determine the oxygen concentration in ambient air. Normally, air contains approximately 20.9% oxygen by volume. Oxygen measurements are of particular importance for work in enclosed spaces, low-lying areas, or in the vicinity of accidents that have produced heavier-than-air vapors which could displace ambient air. The meters are calibrated for sea level and may indicate a false negative (i.e, O_2 content) at higher altitudes. Since the air has been displaced by other substances, these oxygen-deficient areas are also prime locations for taking additional organic vapor and combustible gas measurements. Oxygen-enriched atmospheres increase the potential for fires by their ability to contribute to combustion or to chemically react with flammable compounds and promote auto-ignition.

C Toxic Atmosphere Analyzers

The toxic atmosphere analyzer is a compound-specific instrument, designed and calibrated to identify and quantify a specific compound or class of compounds in either gaseous or vapor form. Cross-sensitivity to air pollutants not of interest may be lead to erroneous results.

U.S. EPA/ERT has the following toxic atmosphere analyzers: carbon monoxide, phosgene, nitrous oxide, hydrogen cyanide, sulfur dioxide, hydrogen sulfide, and chlorine gas.

C Aerosol/Particulate Monitors

A Real-Time Aerosol/Particulate Monitor (RAM) displays readings for total particulates. The instrument employs a pulse light emitting diode which generates a narrow band emission in conjunction with a photovoltaic cell to detect light scattered from particulates.

The U.S. EPA/ERT uses the RAM when the contaminant of concern is associated with particulates, and when responding to fires involving hazardous materials, to identify plume levels. The instrument is very useful in determining the presence of a plume when it is not visible. The U.S. EPA/ERT typically uses RAMs on tripods to obtain particulate concentrations at the breathing zone level. Personal dataloggers are used with the RAMs to document minimum, average and maximum concentrations. This provides real-time data without requiring those in personal protective equipment to be constantly present in the plume.

C Chemical Detector Tubes (Colorimetric Tubes)

A chemical detector tube is a hollow, tube-shaped, glass body containing one or more layers of chemically impregnated inert material. To use, the fused ends are broken off and a manufacturer-specified volume of air is drawn through the tube with a pump to achieve a given detection limit. The chemicals contained within the packing material undergo a chemical reaction with the airborne pollutant present, producing a color change during the intake of each pump stroke. The concentration of a pollutant is indicated by the length of discoloration on a calibrated scale printed on the detector tube.

C Radiation Meters

Radiation meters determine the presence and level of radiation. The meters use a gas or solid ion detection media which becomes ionized when radiation is present. The meters are normally calibrated to one probe. Meters that detect alpha, beta, and gamma radiation are available.

C Gold Film (Hydrogen Sulfide and Mercury Vapor) Monitors

Hydrogen sulfide (H_2S) and Mercury (Hg) monitors operate on the principle that electric resistivity increases across a gold film as a function of H_2S and Hg concentration. The monitors provide rapid and relatively low detection limits for H_2S and Hg in air. After extensive sampling periods or high concentrations of H_2S and Hg, the gold film must be heated to remove contamination and return the monitor to its original sensitivity.

C Infrared Detectors

Infrared detectors such as the Miniature Infrared Analyzer (MIRAN) use infrared (IR) absorption as a function of specific compounds. MIRAN instruments apply to situations where the contaminants are identified but concentrations are not. MIRAN instruments generally require AC power.

SPECIALIZED ANALYTICAL INSTRUMENTS

The continuous monitors described above provide qualitative measurement of air contaminants. Quantitative measurements in the field can be obtained using more sophisticated instruments, such as portable Gas Chromatographs, to analyze grab samples.

C Direct Air Sampling Portable Gas Chromatographs (GCs)

Portable GCs use gas chromatography to identify and quantify compounds. The time it takes for a compound to move through a chromatographic column is a function of that specific compound or group of compounds. A trained technician with knowledge of the range of expected concentrations of compounds can utilize a portable GC in the field to analyze grab samples. GCs generally require AC power and shelter to operate. This method is limited by its reliance on a short-term grab sample to be representative of the air quality at a site.

- Remote Optical Sensing

  This technique, also referred to as long-path or open-path monitoring, involves transmitting either an infrared or ultraviolet light beam across a long open path and measuring the absorbance at specific wavelengths. The technique is capable of analyzing any preselected organic or inorganic volatile compound that can be resolved from compounds naturally occurring in ambient air. Current projected removal applications include perimeter monitoring during site cleanups and measurement of emission source strengths during site assessments.

- TAGA Direct Air Sampling Mass Spectrometer/Mass Spectrometer

  The Trace Atmospheric Gas Analyzer (TAGA), which is operated by the U.S. EPA/ERT, is capable of real-time detection of preselected organic compounds at low parts-per-billion concentrations. The instrument has been successfully used by the U.S. EPA/ERT for isolating individual emission plumes and tracking those plumes back to their sources.

APPENDIX B

Air Sampling Equipment and Media/Devices

AIR SAMPLING EQUIPMENT

C High-Volume, Total Suspended Particulate (TSP) Samplers

High-volume TSP samplers collect all suspended particles by drawing air across an 8- by 10-inch glass-quartz filter. The sample rate is adjusted to 40 cubic feet per minute (CFM), or 1134 liters per minute (L/min), and it is held constant by a flow controller over the sample period. The mass of TSPs can be determined by weighing the filter before and after sampling. The composition of the filter varies according to the analytical method and the detection limit required.

C PM-10 Samplers

PM-10 samplers collect particulates with a diameter of 10 microns or less from ambient air. Particulates of this size represent the respirable fraction, and thus are of special significance. PM-10 samplers can be high-volume or low-volume. The high-volume sampler operates in the same manner as the TSP sampler at a constant flow rate of 40 CFM; it draws the sample through a special impactor head which collects particulates of 10 microns or less. The particulate is collected on an 8- by 10-inch filter. The low-volume sampler operates at a rate of approximately 17 L/min. The flow must remain constant through the impactor head to maintain the 10-micron cut-off point. The low-volume PM-10 collects the sample on 37-mm Teflon filters.

C High-Volume PS-1 Samplers

High-volume PS-1 samplers draw a sample through polyurethane foam (PUF) or a combination foam and XAD-2 resin plug, and a glass quartz filter at a rate of 5-10 CFM (144 to 282 L/min). This system is excellent for measuring low concentrations of semivolatiles, PCBs, pesticides, or chlorinated dioxins in ambient air.

C Area Sampling Pumps

These pumps provide flow-rate ranges of 2-20 L/min and have a telescopic sampling mast with the sampling train. Because of the higher volume, this pump is suitable for sampling low concentrations of airborne contaminants (i.e., asbestos sampling). These pumps are also used for metals, pesticides and PAH sampling which require large sample volumes.

C Personal Sampling Pumps

Personal sampling pumps are reliable portable sampling devices that draw air samples through a number of sampling media including resin tubes, impingers, and filters. Flow rates are usually adjustable from 0.1 to 4 L/min (or 0.01 to .75 L/min with a restrictive orifice) and can remain constant for up to 8 hours on one battery charge or continuously with an AC charger/converter.

C Canister Samplers

Evacuated canister sampling systems use the pressure differential between the evacuated canister and ambient pressure to bleed air into the canister. The sample is bled into the canister at a constant rate over the sampling period using a critical orifice, a mechanically compensated regulator, or a mass flow control
device until the canister is near atmospheric pressure.

Pressure canister sampling systems use a pump to push air into the canister. To maintain a higher, more controlled flow, the pump typically controls the pressure differential across a critical orifice at the

inlet of the canister, resulting in a pressurized canister at the completion of sampling.

AIR SAMPLING MEDIA/DEVICES

If possible, before employing a specific sampling method, consult the laboratory that will conduct the analyses. Many of the methods can be modified to provide better results or a wider range of results.

C Summa^{R} Canisters

Summa canisters are highly polished passivated stainless steel cylinders. The Summa polishing process brings chrome and nickel to the surface of the canisters, which results in an inert surface. This surface restricts adsorption or reactions that occur on the canister's inner surface after collection. At the site, the canister is either placed in a sampler to control sample collection rate, or opened to collect a grab sample. Samples can be collected by allowing air to bleed into or be pumped into the canister. U.S. EPA/ERT uses 6-liter Summa canisters for VOC and permanent gas analysis.

C Passive Dosimeters

Passive dosimeters are clip-on vapor monitors (samplers) in which the diffused contaminants are absorbed on specially prepared active surfaces. Industrial hygienists commonly use dosimeters to obtain time-weighted averages or concentrations of chemical vapors, as they can trap over 130 organic compounds. Selective dosimeters have also been developed for a number of chemicals including formaldehyde, ethylene oxide, hydrogen sulfide, mercury vapor, nitrogen dioxide, sulfur dioxide, and ozone. Dosimeters must be sent to a laboratory for analysis.

C Polyurethane Foam (PUF)

PUF is a sorbent used with a glass filter for the collection of semivolatile organic compounds such as pesticides, PCBs, chlorinated dioxins and furans, and PAHs. Fewer artifacts (chemical changes that occur to collected compounds) are produced than with some other solid sorbents. PUF is used with the PS-1 sampler and U.S. EPA Method TO13. PUF can also be used with personal sampling pumps when sampling for PAHs using the Lewis/McCloud method. Breakthrough of the more volatile PCBs and PAHs may occur when using PUF.

C Sampling Bags (Tedlar^{R})

Sampling bags, like canisters, transport air samples to the laboratory for analysis. Samples are generally pumped into the bags, but sometimes a lung system is used, in which a pump creates a vacuum around the bag in a vacuum box. Then the sample flows from a source into the bag. This method is used for VOCs, fixed gases (CO_2, O_2, and N_2) and methane.

C Impingers

An impinger allows an air sample to be bubbled through a solution, which collects a specific contaminant by either chemical reaction or absorption. For long sampling periods, the impinger may need to be kept in an ice bath to prevent the solution from evaporating during sampling. The sample is drawn through the impinger by using a sampling pump or more elaborate sampling trains with multiple impingers.

C Sorbent Tubes/Cartridges

A variety of sampling media are available in sorbent tubes, which are used primarily for industrial hygiene. A few examples are carbon cartridges, carbon molecular sieves, Tenax tubes and tube containing the XAD-2 polymer. Depending upon the sorbent material, tubes can be analyzed using either a solvent extraction or thermal desorption. The former technique uses standard laboratory equipment and allows for multiple analyses of the same sample. The latter technique requires special, but readily available, laboratory equipment and allows only one analysis per sample. In addition, thermal desorption typically allows for lower detection limits by two or more orders of magnitude. Whenever sorbent tubes are

being used for thermal desorption, they should be certified as "clean" by the laboratory doing the analysis.

Thermally Desorbed Media

During thermal desorption, high-temperature gas streams are used to remove the compounds collected on a sorbent medium. The gas stream is injected and often cryofocused into an analytical instrument, such as a GC, for compound analysis:

C Tenax Tubes

Tenax tubes are made from commercially available polymer (p-phenylene oxide) packed in glass or stainless steel tubes through which air samples are drawn or sometimes pumped. These tubes are used in U.S. EPA Method TO1 and VOST for volatile nonpolar organic, some polar organic, and some of the more volatile semivolatile organics. Tenax is not appropriate for many of the highly volatile organics (with vapor pressure greater than approximately 200 mm Hg).

C Carbonized Polymers

The carbonized molecular sieve (CMS), a carbonized polymer, is a commercially available, carbon sorbent packed in stainless-steel sampling tubes through which air samples are drawn or sometimes pumped. These are used in U.S. EPA Method TO2 for highly volatile nonpolar compounds which have low-breakthrough volumes on other sorbents. When high-thermal desorption temperatures are used with CMS, more variability in analysis may occur than with other sorbents.

C Mixed Sorbent Tubes

Sorbent tubes can contain two type of sorbents. Combining the advantages of each sorbent into one tube increases the possible types of compounds to be sampled. The combination of two sorbents can also reduce the chance that highly volatile compounds will break through the sorbent media. An example of a mixed sorbent tube is the combination of Tenax and charcoal with a carbonized molecular sieve. A potential problem with mixed sorbent tubes is the breakthrough of a compound from an earlier sorbent to a later sorbent from which it cannot be desorbed.

Solvent-Extracted Media

Solvent-extracted media use the principle of chemical extraction to remove compounds collected on a sorbent media. The chemical solvent is injected into an instrument, such as a GC, for analysis of compounds. Examples of solvent-extracted media follow:

C Chemically Treated Silica Gel

Silica gel is a sorbent which can be treated with various chemicals. The chemically treated silica gel can then be used to sample for specific compounds in air. Examples include the DNPH-coated silica gel cartridge used with U.S. EPA Method TO11.

C XAD-2 Polymers

XAD-2 polymers usually are placed in tubes, custom-packed sandwich-style with polyurethane foam, and prepared for use with U.S. EPA Method TO13 or the semi-VOST method. The polymers are used for the collection of semivolatile polar and nonpolar organic compounds. The compounds collected on the XAD-2 polymer are chemically extracted for analysis.

C Charcoal Cartridges

Charcoal cartridges, consisting of primary and backup sections, trap compounds by adsorption. Ambient air is drawn through them so that the backup section verifies that breakthrough of the analytes on the first section did not occur, and the sample collection was therefore quantitative. Quantitative sample collection is evident by the presence of target chemicals on the first charcoal section and the absence on the second section. Next, the adsorbed compounds must be eluted, usually with a solvent extraction, and analyzed by GC with a detector, such as a Mass Spectrometer (MS).

C Tenax Tubes

Cartridges are used in OSHA and NIOSH methods in a manner similar to charcoal cartridges but typically for less volatile compounds.

Particulate Filters

Particulate filters are used by having a sampling pump pass air through them. The filter collects the particulates present in the air and is then analyzed for particulate mass or chemical or radiological composition. Particulate filters are made from different materials which are described below.

C Mixed Cellulose Ester (MCE)

MCE is manufactured from mixed esters of cellulose which are a blend of nitro-cellulose and cellulose acetate. MCE filters are used often for particulate sampling.

C Glass Fiber

Glass fiber is manufactured from glass fibers without a binder. Particulate filters with glass fiber provide high flow rates, wet strength, and high, solid holding capacity. Generally, the filters are used for gravimetric analysis of particulates.

C Polyvinyl Chloride

Particulate filters with polyvinyl chloride are resistant to concentrated acids and alkalis. Their low moisture pickup and light tare weight make them ideal for gravimetric analysis.

C Teflon

Teflon is manufactured from polytetrafluorethylene (PTFE). Particulate filters with Teflon are easy to handle and exceptionally durable. Teflon filters are used for metal collection.

C Silver

Particulate filters manufactured from pure silver have high collection efficiency and uniform pore size. These filters are used for mercury collection and analysis.

C Cellulose

Particulate filters with cellulose contain less than 0.01% ash. These filters are used to collect particulates.

CHIP, WIPE, AND SWEEP SAMPLING

SOP#: 2011
DATE: 11/16/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) outlines the recommended protocol and equipment for collection of representative chip, wipe, and sweep samples to monitor potential surficial contamination.

This method of sampling is appropriate for surfaces contaminated with non-volatile species of analytes (i.e., PCB, PCDD, PCDF, metals, cyanide, etc.) Detection limits are analyte specific. Sample size should be determined based upon the detection limit desired and the amount of sample requested by the analytical laboratory. Typical sample area is one square foot. However, based upon sampling location, the sample size may need modification due to area configuration.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Since surface situations vary widely, no universal sampling method can be recommended. Rather, the method and implements used must be tailored to suit a specific sampling site. The sampling location should be selected based upon the potential for contamination as a result of manufacturing processes or personnel practices.

Chip sampling is appropriate for porous surfaces and is generally accomplished with either a hammer and chisel, or an electric hammer. The sampling device should be laboratory cleaned and wrapped in clean, autoclaved aluminum foil until ready for use. To collect the sample, a measured and marked off area is chipped both horizontally and vertically to an even depth of 1/8 inch. The sample is then transferred to the proper sample container.

Wipe samples are collected from smooth surfaces to indicate surficial contamination; a sample location is measured and marked off. While wearing a new pair of surgical gloves, a sterile gauze pad is opened, and soaked with solvent. The solvent used is dependent on the surface being sampled. This pad is then stroked firmly over the sample surface, first vertically, then horizontally, to ensure complete coverage. The pad is then transferred to the sample container.

Sweep sampling is an effective method for the collection of dust or residue on porous or non-porous surfaces. To collect such a sample, an appropriate area is measured off. Then, while wearing a new pair of disposable surgical gloves, a dedicated brush is used to sweep material into a dedicated dust pan. The sample is then transferred to the proper sample container.

Samples collected by all three methods are then sent to the laboratory for analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples should be stored out of direct sunlight to reduce photodegredation, cooled to 4°C and shipped to the laboratory performing the analysis. Appropriately sized laboratory cleaned, glass sample jars should be used for sample collection. The amount of sample required will be determined in concert with the analytical laboratory.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

This method has few significant interferences or problems. Typical problems result from rough porous

surfaces which may be difficult to wipe, chip, or sweep.

5.0 EQUIPMENT

Equipment required for performing chip, wipe, or sweep sampling is as follows:

- Lab clean sample containers of proper size and composition
- Site logbook
- Sample analysis request forms
- Chain of Custody records
- Custody seals
- Field data sheets
- Sample labels
- Disposable surgical gloves
- Sterile wrapped gauze pad (3 in. x 3 in.)
- Appropriate pesticide (HPLC) grade solvent
- Medium sized laboratory cleaned paint brush
- Medium sized laboratory cleaned chisel
- Autoclaved aluminum foil
- Camera
- Hexane (pesticide/HPLC grade)
- Iso-octane
- Distilled/deionized water

6.0 REAGENTS

Reagents are not required for preservation of chip, wipe or sweep samples. However, reagents will be utilized for decontamination of sampling equipment.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or preclean equipment, and ensure that it is in working order.

4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Mark all sampling locations. If required the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Chip Sample Collection

Sampling of porous surfaces is generally accomplished by using a chisel and hammer or electric hammer. The sampling device should be laboratory cleaned or field decontaminated as per the Sampling Equipment Decontamination SOP. It is then wrapped in cleaned, autoclaved aluminum foil. The sampler should remain in this wrapping until it is needed. Each sampling device should be used for only one sample.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.

2. Record surface area to be chipped.

3. Don a new pair of disposable surgical gloves.

4. Open a laboratory-cleaned chisel or equivalent sampling device.

5. Chip the sample area horizontally, then vertically to an even depth of approximately 1/8 inch.

6. Place the sample in an appropriately prepared sample container with a Teflon lined cap.

7. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site logbook and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.

8. Store samples out of direct sunlight and cool to 4EC.

9. Follow proper decontamination procedures then deliver sample(s) to the laboratory for analysis.

7.3 Wipe Sample Collection

Wipe sampling is accomplished by using a sterile

gauze pad, adding a solvent in which the contaminant is most soluble, then wiping a pre-determined, pre-measured area. The sample is packaged in an amber jar to prevent photodegradation and packed in coolers for shipment to the lab. Each gauze pad is used for only one wipe sample.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.

2. Record surface area to be wiped.

3. Don a new pair of disposable surgical gloves.

4. Open new sterile package of gauze pad.

5. Soak the pad with solvent of choice.

6. Wipe the marked surface area using firm strokes. Wipe vertically, then horizontally to insure complete surface coverage.

7. Place the gauze pad in an appropriately prepared sample container with a Teflon-lined cap.

8. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site logbook and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.

9. Store samples out of direct sunlight and cool to 4ºC.

10. Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

7.4 Sweep Sample Collection

Sweep sampling is appropriate for bulk contamination. This procedure utilizes a dedicated, hand held sweeper brush to acquire a sample from a pre-measured area.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.

2. Record the surface area to be swept.

3. Don new pair of disposable surgical gloves.

4. Sweep the measured area using a dedicated brush; collect the sample in a dedicated dust pan.

5. Transfer sample from dust pan to sample container.

6. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site log book and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.

7. Store samples out of direct sunlight and cool to 4EC.

8. Leave contaminated sampling device in the sample material, unless decontamination is practical.

9. Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

8.0 CALCULATIONS

Results are usually provided in mg/g, µg/g, mass per unit area, or other appropriate measurement. Calculations are typically done by the laboratory.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following general quality assurance procedures apply:

1. All data must be documented on standard chain of custody forms, field data sheets or within the site logbook.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

The following specific quality assurance activities apply to wipe samples:

For wipe samples, a blank should be collected for each sampling event. This consists of a sterile gauze pad, wet with the appropriate solvent, and placed in a prepared sample container. The blank will help identify potential introduction of contaminants via the sampling methods, the pad, solvent or sample container. Spiked wipe samples can also be collected to better assess the data being generated. These are prepared by spiking a piece of foil of known area with a standard of the analyte of choice. The solvent containing the standard is allowed to evaporate, and the foil is wiped in a manner identical to the other wipe samples.

Specific quality assurance activities for chip and sweep samples should be determined on a site specific basis.

10.0 DATA VALIDATION

A review of the quality control samples will be conducted and the data utilized to qualify the environmental results.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow EPA, OSHA and corporate health and safety procedures.

12.0 REFERENCES

U.S. EPA, A Compendium of Superfund Field Operation Methods. EPA/540/5-87/001.

NJDEP Field Sampling Procedures Manual, February, 1988.

U. S. EPA ENVIRONMENTAL RESPONSE TEAM

STANDARD OPERATING PROCEDURES

SOIL SAMPLING

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SUPERCEDES: SOP #2012; Revision 0.0; 11/16/94; U.S. EPA Contract 68-C4-0022.

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative soil samples. Sampling depths are assumed to be those that can be reached without the use of a drill rig, direct-push, or other mechanized equipment (except for a back-hoe). Analysis of soil samples may determine whether concentrations of specific pollutants exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or, if required, a backhoe.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is not generally recommended. Samples should, however, be cooled and protected from sunlight to minimize any potential reaction. The amount of sample to be collected and proper sample container type are discussed in ERT/REAC SOP #2003 Rev. 0.0 08/11/94, *Sample Storage, Preservation and Handling.*

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary potential problems associated with soil sampling - cross contamination of samples and improper sample collection. Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

5.0 EQUIPMENT

Soil sampling equipment includes the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s)
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
  - Tubes
  - Points
  - Drive head
  - Drop hammer
  - Puller jack and grip
- Backhoe

6.0 REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in ERT/REAC SOP #2006 Rev. 0.0 08/11/94, *Sampling Equipment Decontamination*, and the site specific work plan.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order.

4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations should be utility-cleared by the property owner or the On-Scene-Coordinator (OSC) prior to soil sampling; and utility clearance should always be confirmed before beginning work.

7.2 Sample Collection

7.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample.

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required. Tools plated with chrome or other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

The following procedure is used to collect surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.

2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.

5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler, and unscrew the drill rods.

8. Remove the cutting tip and the core from the device.

9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.

10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly.

    When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

12. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

7.2.3 Sampling with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth.

The following procedure is used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix A) into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should

be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils".

The following procedures are used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.

4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.

5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.

6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

7.2.5 Test Pit/Trench Excavation

A backhoe can be used to remove sections of soil, when detailed examination of soil characteristics are required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation.

The following procedures are used for collecting soil samples from test pits or trenches:

1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities.

2. Review the site specific Health & Safety plan and ensure that all safety precautions including appropriate monitoring equipment are installed as required.

3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than five feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.

4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done.

5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket.

6. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration

activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OHSA and corporate health and safety procedures, in addition to the procedures specified in the site specific Health & Safety Plan..

12.0 REFERENCES

Mason, B.J. 1983. Preparation of Soil Sampling Protocol: Technique and Strategies. EPA-600/4-83-020.

Barth, D.S. and B.J. Mason. 1984. Soil Sampling Quality Assurance User's Guide. EPA-600/4-84-043.

U.S. Environmental Protection Agency. 1984 Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA-600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. 1980. Samplers and Sampling Procedures for Hazardous Waste Streams. EPA-600/2-80-018.

ASTM D 1586-98, ASTM Committee on Standards, Philadelphia, PA.

APPENDIX A
Figures
SOP #2012
February 2000

FIGURE 1. Sampling Augers

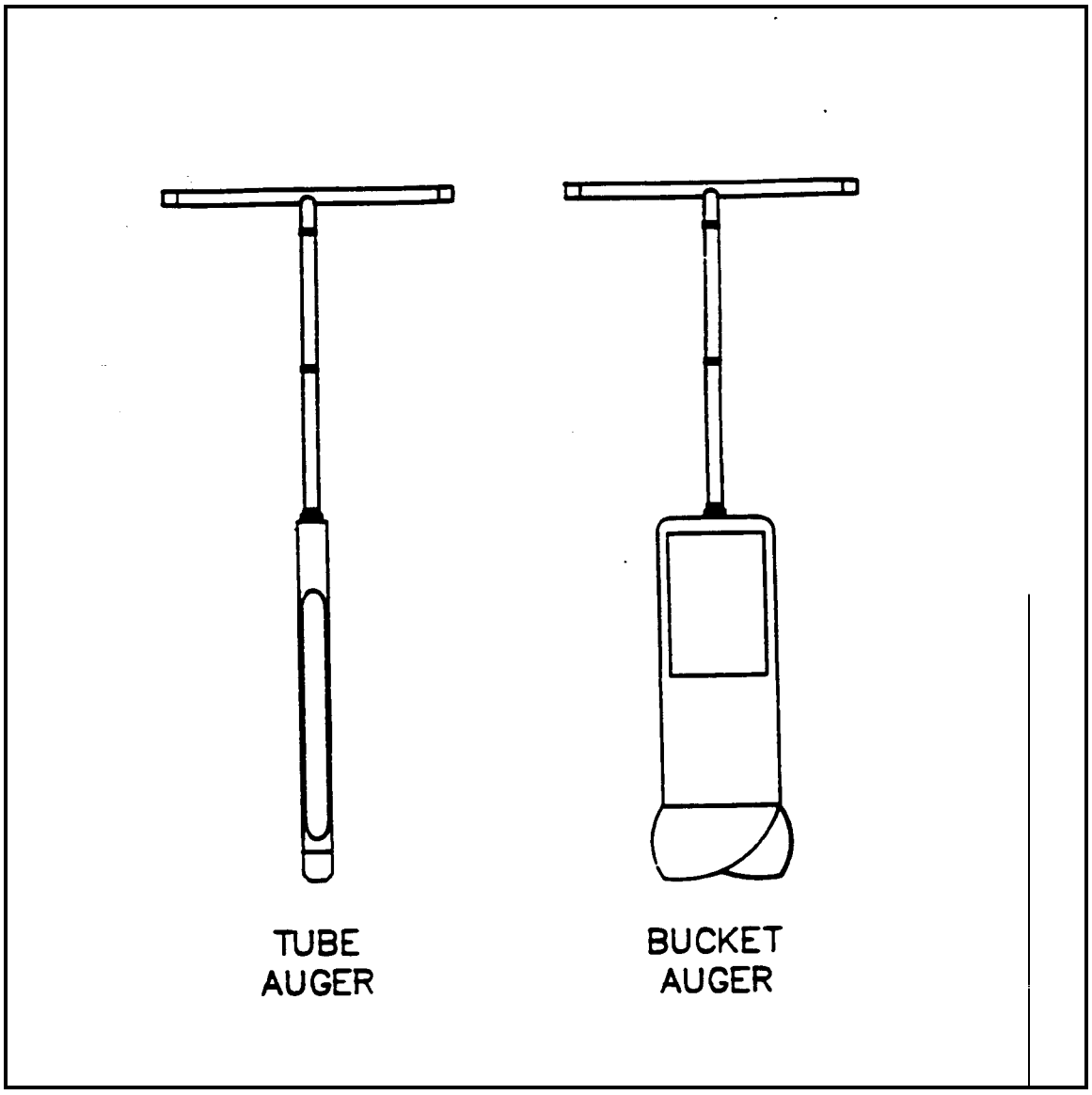
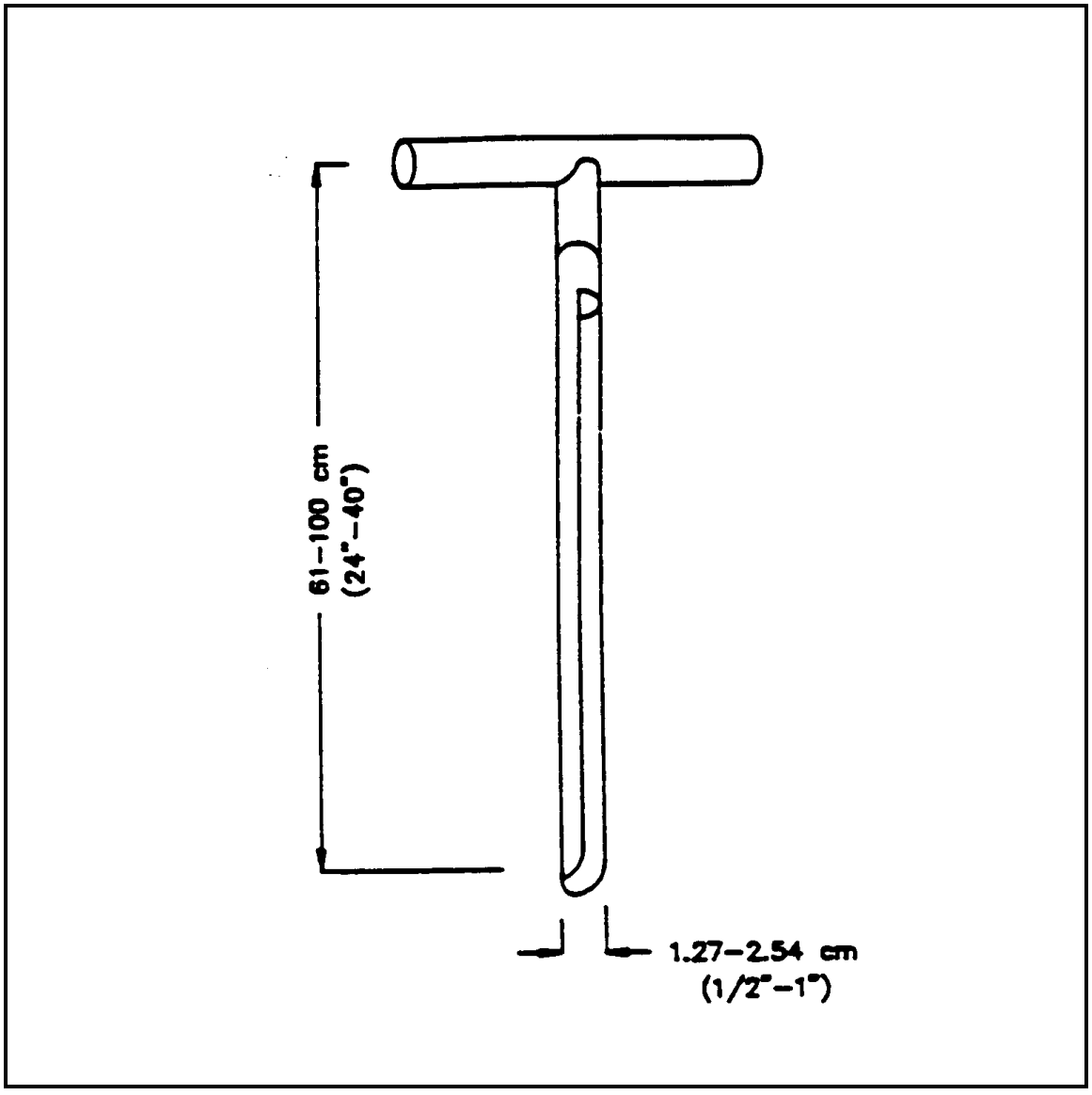


FIGURE 2. Sampling Trier



ASBESTOS SAMPLING

SOP#: 2015
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method for TEM[2]; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)[3]. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)[4] and its addendum 40 CFR 763 (October 30, 1987)[4] provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 μm[5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and

medical surveillance[5,6].

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.

- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.

- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.

- There is increased opportunity for fiber loss or introduction of extraneous contamination.

- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2 µm mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m^3).

	Concentration	Flow Rate
C Low RAM readings:	<6.0 mg/m^3	11-15. L/min
C Medium RAM readings:	>6.0 mg/m_3	7.5 L/min
C High RAM readings:	>10. mg/m^3	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump.Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.

3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.

2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.

4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.

(2) Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.

(3) National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.

(4) U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A

Tables

TABLE 1.

SAMPLE STATIONS FOR OUTDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Upwind/Background[1]	Collect a minimum of two simultaneous upwind/background samples 30º apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

APPENDIX A (Cont'd)

Tables

TABLE 2

SAMPLE STATIONS FOR INDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

FIGURE 1. Transmission Electron Microscopy Filter Cassette

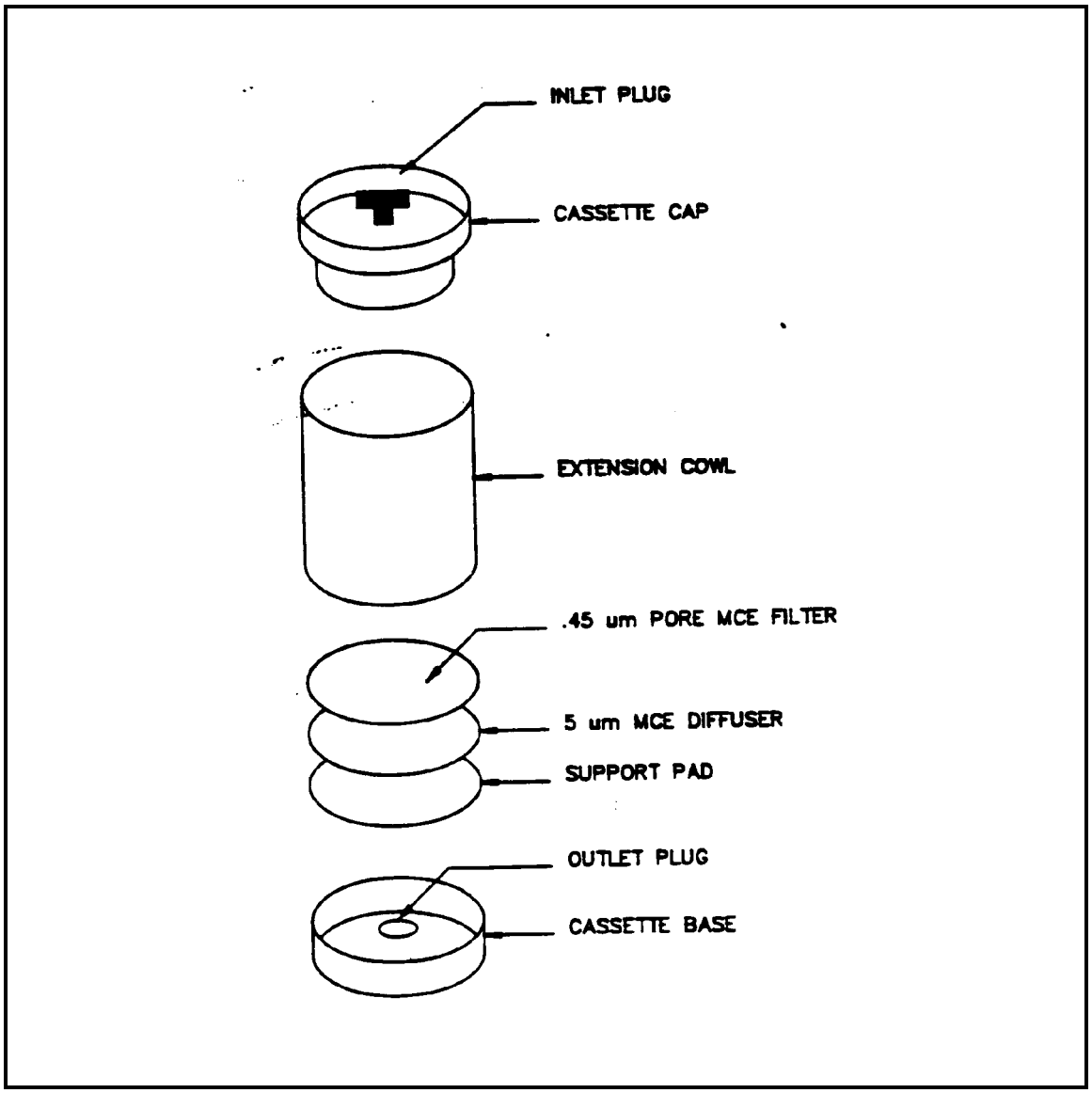


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FIGURE 2. Phase Contrast Microscopy Filter Cassette

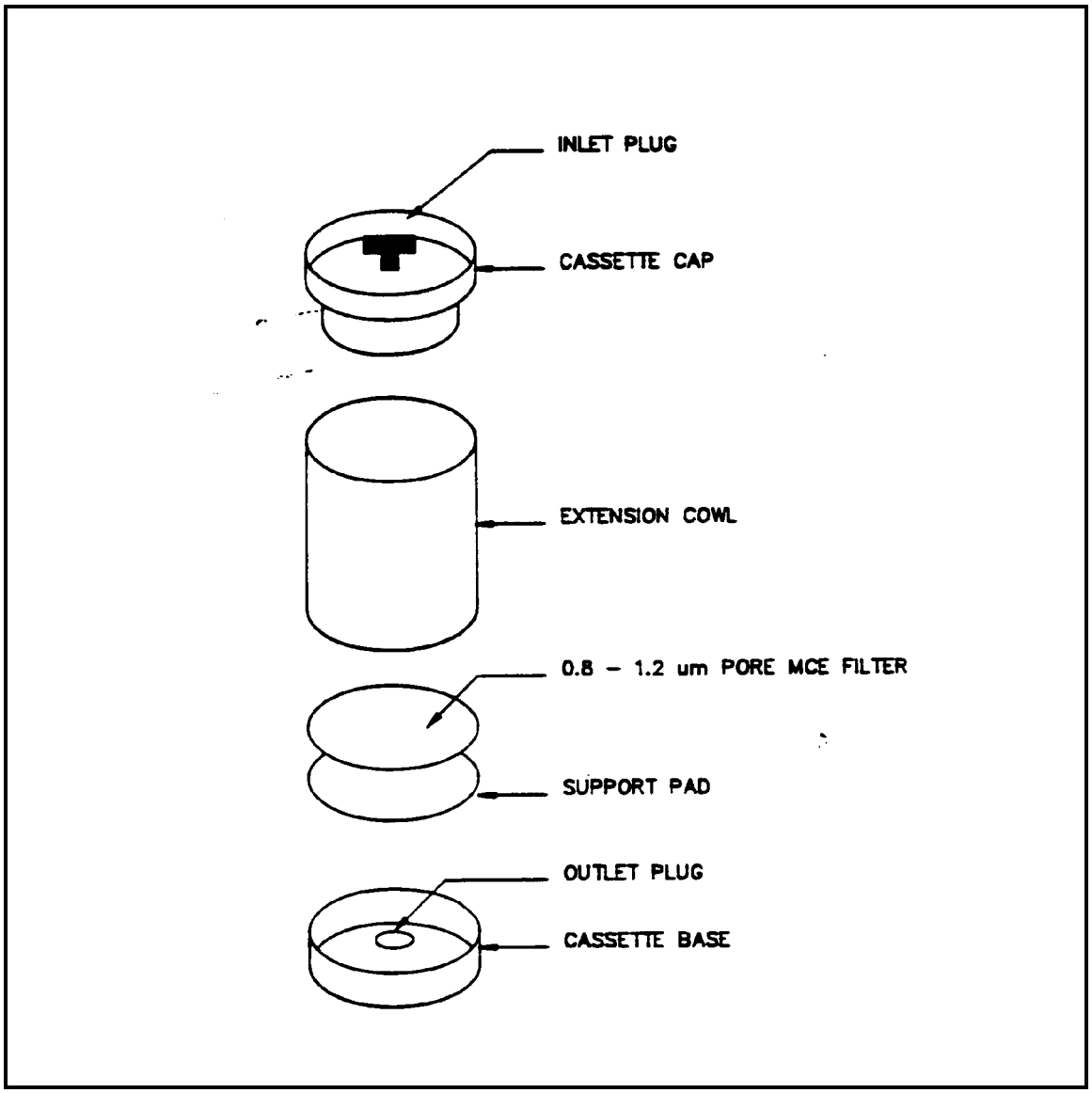

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FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter

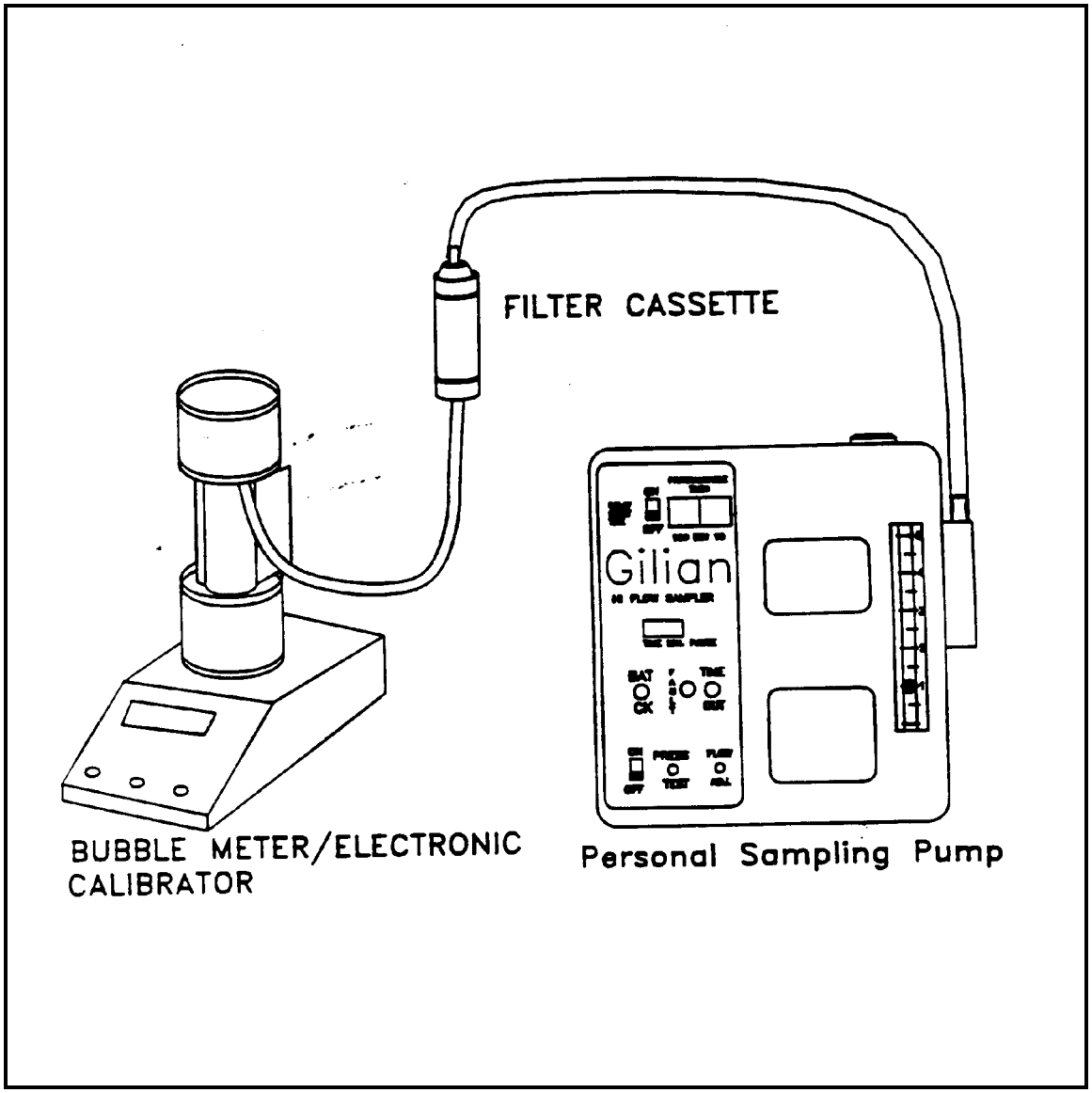

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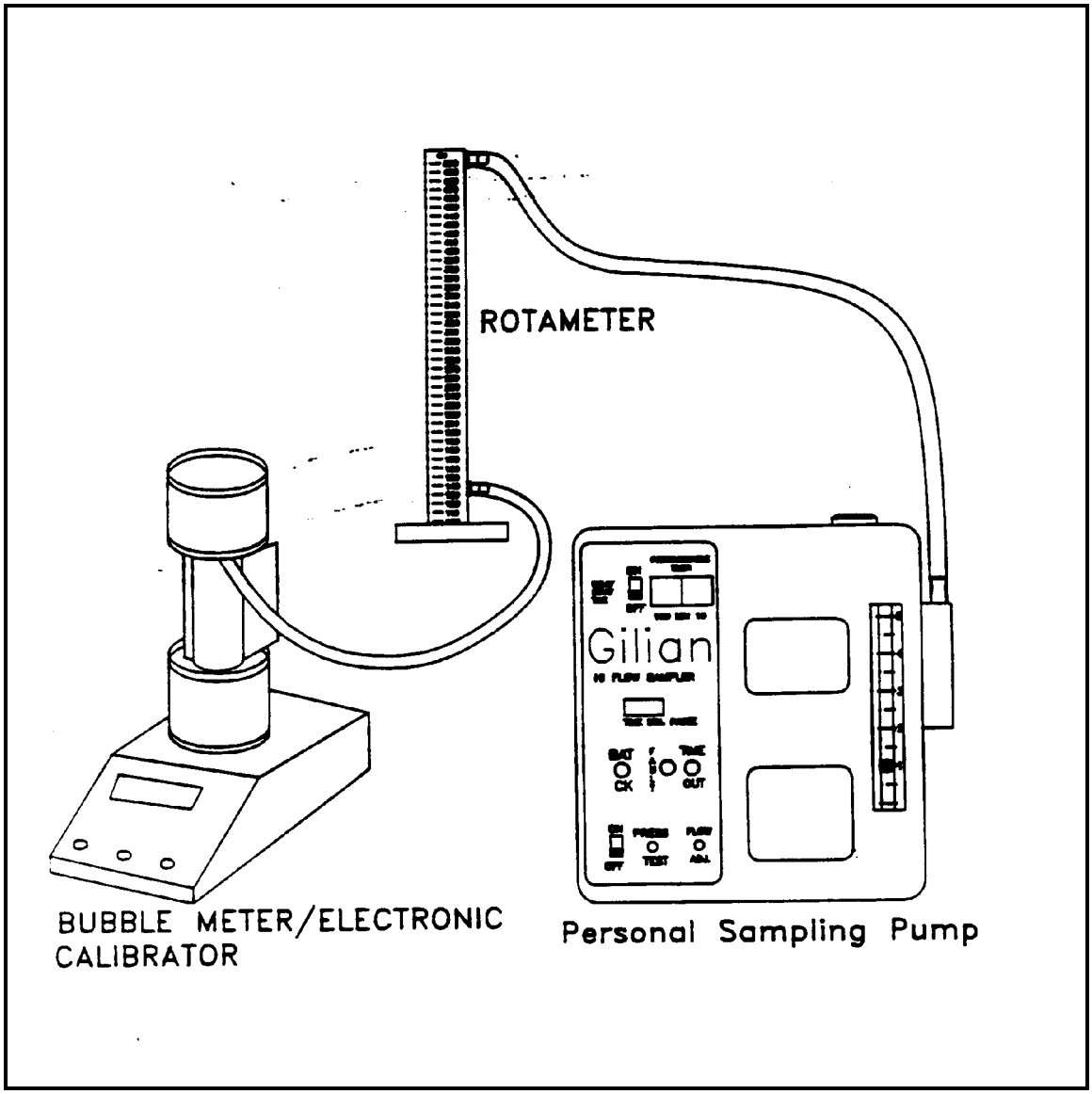
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



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Figures

FIGURE 5. Calibrating a Sampling Pump with a Rotameter

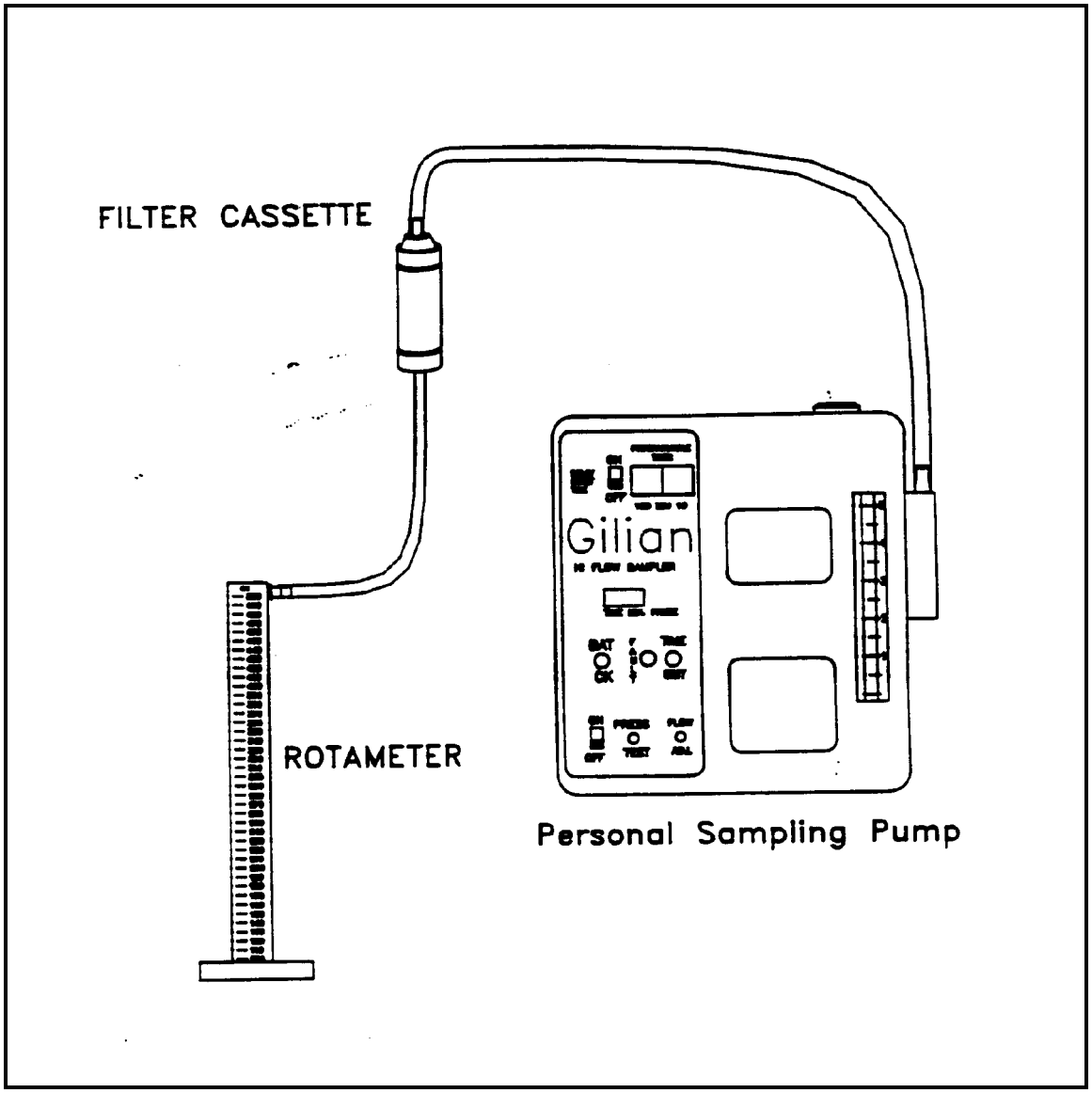

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FIGURE 6. Personal Sampling Train for Asbestos

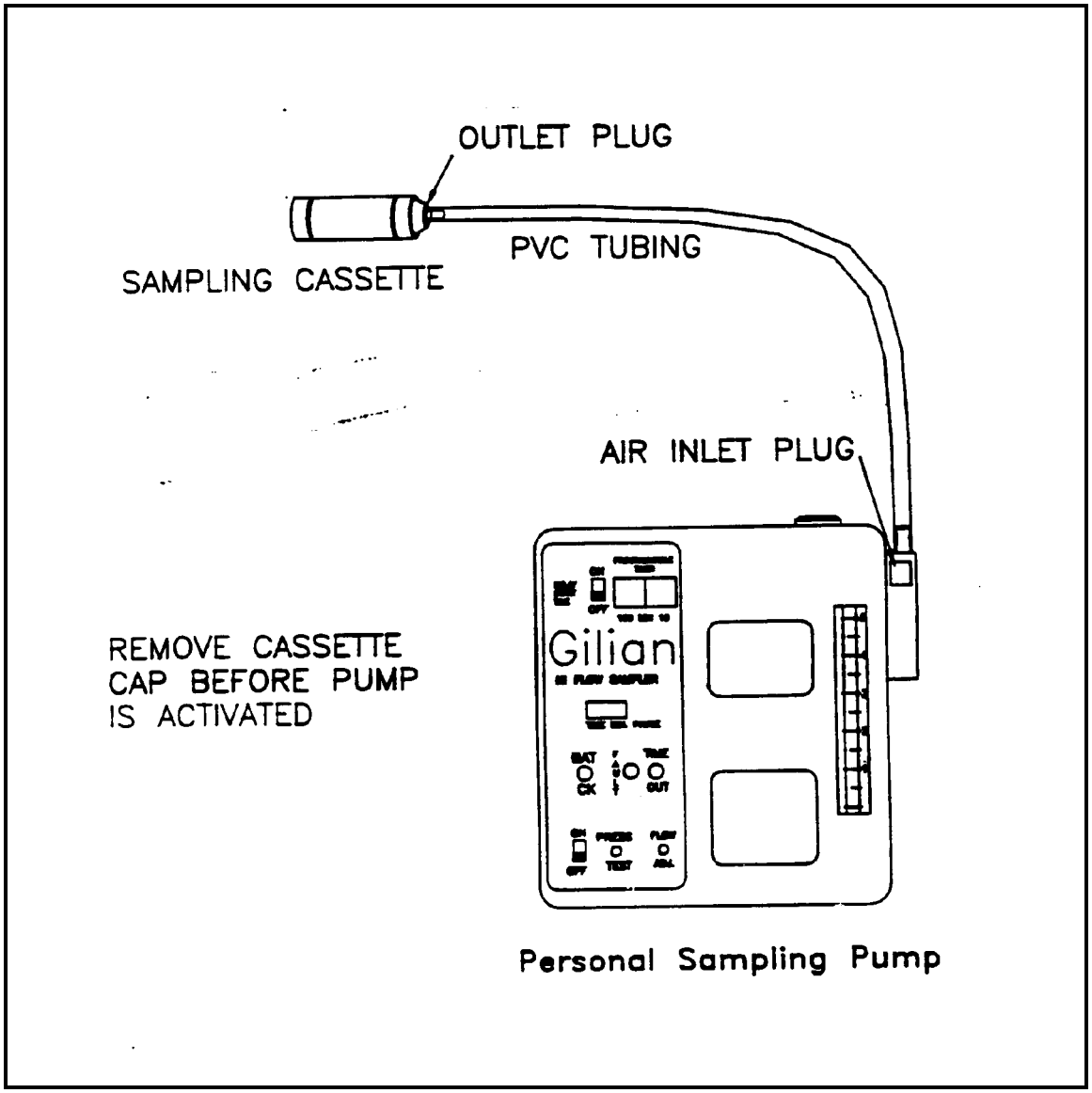

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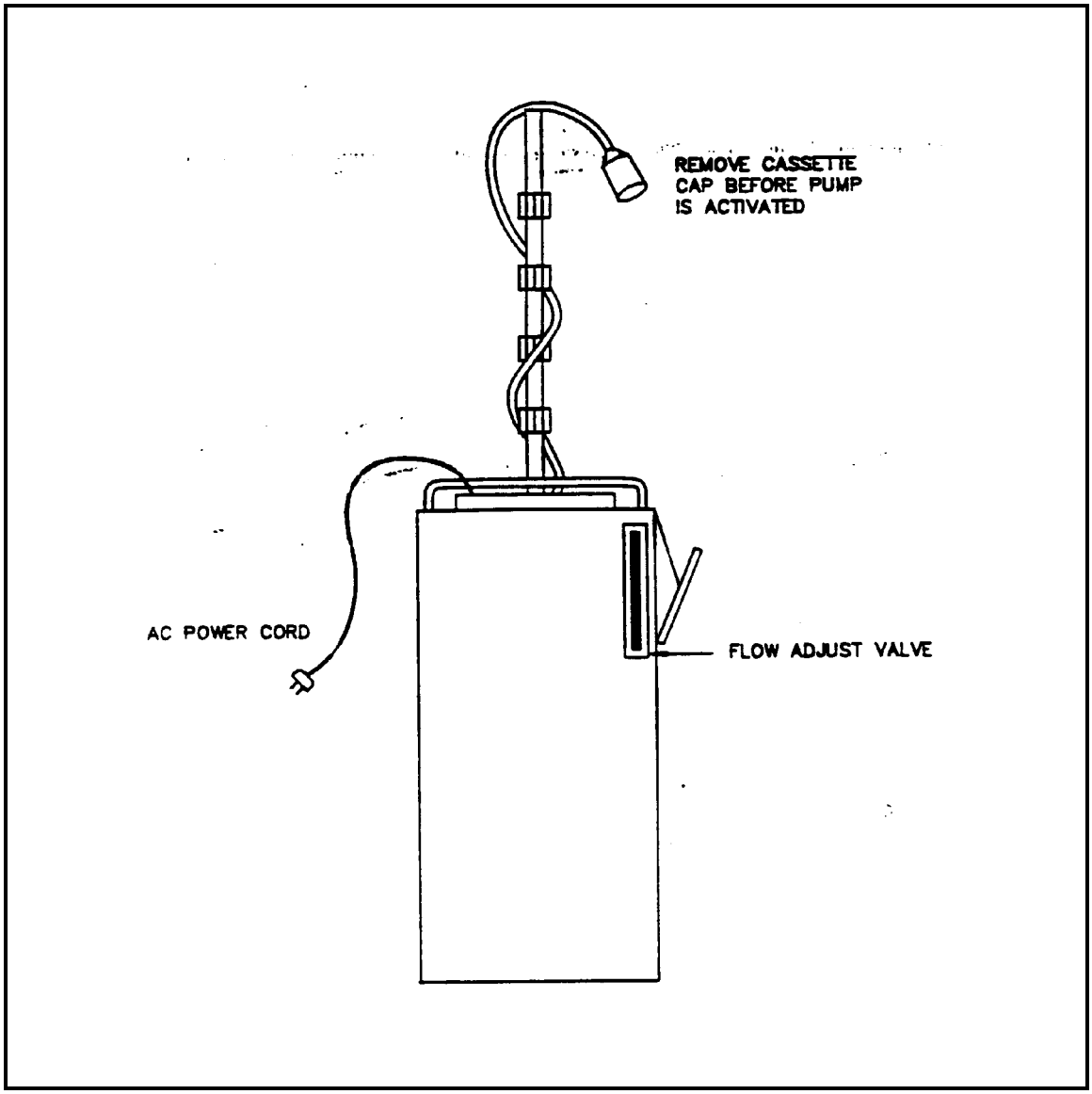
FIGURE 7. High Flow Sampling Train for Asbestos



ATTACHMENT C

ANALYTICAL METHODS

United States
Environmental Protection
Agency

Office of Research and
Development
Washington, DC 20460

EPA/600/R-93/116
July 1993

EPA

Test Method

Method for the Determination of Asbestos in Bulk Building Materials

EPA/600/R-93/116
July 1993

TEST METHOD

METHOD FOR THE DETERMINATION OF ASBESTOS IN BULK BUILDING MATERIALS

by

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DISCLAIMER

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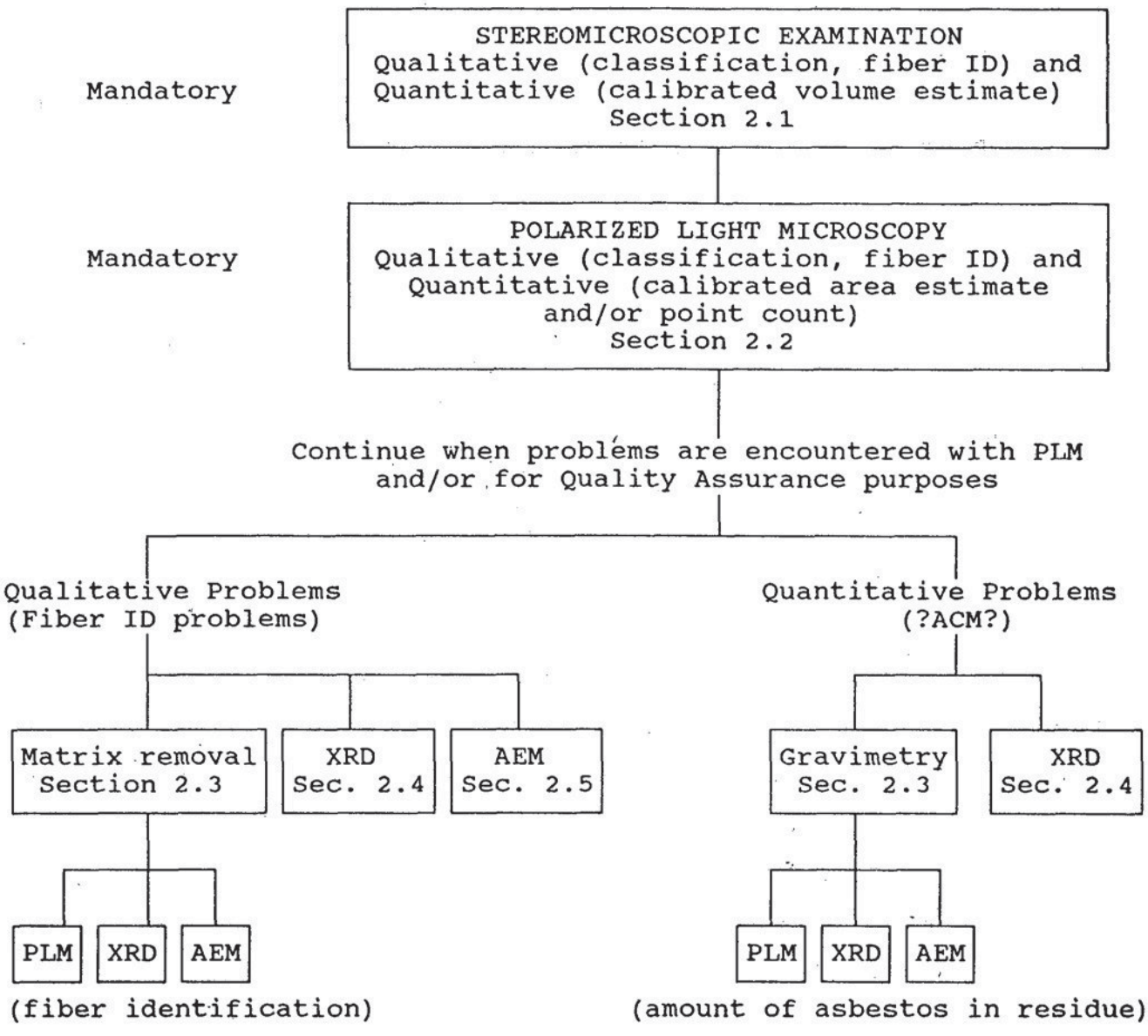
1.0 INTRODUCTION

Laboratories are now called upon to identify asbestos in a variety of bulk building materials, including loose-fill insulations, acoustic and thermal sprays, pipe and boiler wraps, plasters, paints, flooring products, roofing materials and cementitious products.

The diversity of bulk materials necessitates the use of several different methods of sample preparation and analysis. An analysis with a simple stereomicroscope is always followed by a polarized light microscopic (PLM) analysis. The results of these analyses are generally sufficient for identification and quantitation of major concentrations of asbestos. However, during these stereomicroscopic and PLM analyses, it may be found that additional techniques are needed to: 1) attain a positive identification of asbestos; 2) attain a reasonable accuracy for the quantity of asbestos in the sample; or 3) perform quality assurance activities to characterize a laboratory's performance. The additional techniques include x-ray diffraction (XRD), analytical electron microscopy (AEM), and gravimetry, for which there are sections included in the method. Other techniques will be considered by the Environmental Protection Agency (EPA) and may be added at some future time. Table 1-1 presents a simplified flowchart for analysis of bulk materials.

This Method for the Determination of Asbestos in Bulk Building Materials outlines the applicability of the various preparation and analysis methods to the broad spectrum of bulk building materials now being analyzed. This method has been evaluated by the EPA Atmospheric Research and Exposure Assessment Laboratory (EPA/AREAL) to determine if it offers improvements to current analytical techniques for building materials. This method demonstrated a capability for improving the precision and accuracy of analytical results. It contains significant revisions to procedures outlined in the Interim Method,[1] along with the addition of several new procedures. Each technique may reduce or introduce bias, or have some effect on the precision of the measurement, therefore results need to be interpreted judiciously. Data on each technique, especially those new to asbestos analysis, will be collected over time and carefully evaluated, with resulting recommendations for changes to the Method to be passed on to the appropriate program office within EPA.

TABLE 1-1. SIMPLIFIED FLOWCHART FOR ANALYSIS OF BULK MATERIALS



This is an analytical method. It is not intended to cover bulk material sampling, an area addressed previously[2,3,4,5] by the EPA. However, subsampling or sample splitting as it pertains to laboratory analysis procedures in this method, is discussed throughout.

1.1 References

1. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
2. **Asbestos-Containing Materials in School Buildings: A Guidance Document,** Part 1 and 2, U.S. E.P.A./O.T.S NO. C00090, 1979.
3. **Asbestos in Buildings: Simplified Sampling Scheme for Friable Surfacing Materials,** U.S. E.P.A. 560/5-85-030a, 1985.
4. **Guidance for Controlling Asbestos-Containing Materials in Buildings,** U.S. E.P.A. 560/5-85-024, 1985.
5. **Asbestos-Containing Materials in Schools: Final Rule and Notice,** 40 CFR Part 763, October, 1987.

2.0 METHODS

2.1 Stereomicroscopic Examination

A preliminary visual examination using a simple stereomicroscope is mandatory for all samples. A sample should be of sufficient size to provide for an adequate examination. For many samples, observations on homogeneity, preliminary fiber identification and semi-quantitation of constituents can be made at this point. Another method of identification and semi-quantitation of asbestos must be used in conjunction with the stereomicroscopic examination. A description of the suggested apparatus needed for stereomicroscopic examination is given in Appendix B.

The laboratory should note any samples of insufficient volume. A sufficient sample volume is sample-type dependent. For samples such as floor tiles, roofing felts, paper insulation, etc., three to four square inches of the layered material would be a preferred sample size. For materials such as ceiling tiles, loose-fill insulation, pipe insulation, etc., a sample size of approximately one cubic inch ($\sim$ 15cc) would be preferred. For samples of thin-coating materials such as paints, mastics, spray plasters, tapes, etc., a smaller sample

size may be suitable for analysis. Generally, samples of insufficient volume **should be rejected**, and further analysis curtailed until the client is contacted. The quantity of sample affects the sensitivity of the analysis and reliability of the quantitation steps. If there is a question whether the sample is representative due to inhomogeneity, the sample should be rejected, at least until contacting the client to see if: 1) the client can provide more material or 2) the client wishes the laboratory to go ahead with the analysis, but with the laboratory including a statement on the limited sensitivity and reliability of quantitation. If the latter is the case, the report of analysis should state that the client was contacted, that the client decided that the lab should use less material than recommended by the method, and that the client acknowledges that this may have limited the sensitivity and quantitation of the method. At the time the client is contacted about the material, he or she should be informed that a statement reflecting these facts will be placed in the report.

2.1.1 Applicability

Stereomicroscopic analysis is applicable to all samples, although its use with vinyl floor tile, asphaltic products, etc., may be limited because of small asbestos fiber size and/or the presence of interfering components. It does not provide positive identification of asbestos.

2.1.2 Range

Asbestos may be detected at concentrations less than one percent by volume, but this detection is highly material dependent.

2.1.3 Interferences

Detection of possible asbestos fibers may be made more difficult by the presence of other nonasbestos fibrous components such as cellulose, fiber glass, etc., by binder/matrix materials which may mask or obscure fibrous components, and/or by exposure to conditions (acid environment, high temperature, etc.) capable of altering or transforming asbestos.

2.1.4 Precision and Accuracy

The precision and accuracy of these estimations are material dependent and must be determined by the individual laboratory for the percent range involved. These values may be

determined for an individual analyst by the in-house preparation and analysis of standards and the use of error bars, control charts, etc.

The labs should also compare to National Voluntary Laboratory Accreditation Program (NVLAP) proficiency testing samples, if the lab participates in the Bulk Asbestos NVLAP, or to external quality assurance system consensus results such as from proficiency testing programs using characterized materials. However, at this time, consensus values for the quantity of asbestos have been shown to be unreliable. Only proficiency testing materials characterized by multiple techniques should be used to determine accuracy and precision.

2.1.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample handling and examination must be carried out in a HEPA-filtered hood, a class 1 biohazard hood or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.1.5.1 Sample Preparation

No sample preparation should be undertaken before initial stereomicroscopic examination. Distinct changes in texture or color on a stereomicroscopic scale that might denote an uneven distribution of components should be noted. When a sample consists of two or more distinct layers or building materials, each should be treated as a separate sample, when possible. Thin coatings of paint, rust, mastic, etc., that cannot be separated from the sample without compromising the layer are an exception to this case and may be included with the layer to which they are attached. Drying (by heat lamp, warm plate, etc.) of wet or damp samples is recommended before further stereomicroscopic examination and is mandatory before PLM examination. **Drying must be done in a safety hood.**

For nonlayered materials that are heterogeneous, homogenization by some means (mill, blender, mortar and pestle) may provide a more even distribution of sample components. It

may also facilitate disaggregation of clumps and removal of binder from fibers (rarely however, it may mask fibers that were originally discernable).

For materials such as cementitious products and floor tiles, breaking, pulverizing, or grinding may improve the likelihood of exposing fibrous components.

It may be appropriate to treat some materials by dissolution with hydrochloric acid to remove binder/matrix materials. Components such as calcite, gypsum, magnesite, etc., may be removed by this method. For materials found to possess a high organic content (cellulose, organic binders), ashing by means of a muffle furnace or plasma asher (for small, cellulosic samples), or dissolution by solvents may be used to remove interfering material. In either case, it is recommended that matrix removal be tracked gravimetrically.

Additional information concerning homogenization, ashing and acid dissolution may be found in Sections 2.2.5.1 and 2.3.

2.1.5.2 Analysis

Samples should be examined with a simple stereomicroscope by viewing multiple fields of view over the entire sample. The whole sample should be observed after placement in a suitable container (watchglass, weigh boat, etc.) substrate. Samples that are very large should be subsampled. The sample should be probed, by turning pieces over and breaking open large clumps. The purpose of the stereomicroscopic analysis is to determine homogeneity, texture, friability, color, and the extent of fibrous components of the sample. This information should then be used as a guide to the selection of further, more definitive qualitative and quantitative asbestos analysis methods. Homogeneity refers to whether each subsample made for other analytical techniques (e.g. the "pinch" mount used for the PLM analysis), is likely to be similar or dissimilar. Color can be used to help determine homogeneity, whether the sample has become wet (rust color), and to help identify or clarify sample labelling confusion between the building material sampler and the laboratory. Texture refers to size, shape and arrangement of sample components. Friability may be indicated by the ease with which the sample is disaggregated (see definitions in Appendix A) as received by the analyst. This does not necessarily represent the friability of the material as determined by the assessor at the collection site. The relative proportion of fibrous

components to binder/matrix material may be determined by comparison to similar materials of known fibrous content. For materials composed of distinct layers or two or more distinct building materials, each layer or distinct building material should be treated as a discrete sample. The relative proportion of each in the sample should be recorded. The layers or materials should then be separated and analyzed individually. Analysis results for each layer or distinct building material should be reported. If monitoring requirements call for one reported value, the results for the individual layers or materials should always be reported along with the combined value. Each layer or material should be checked for homogeneity during the stereomicroscopic analysis to determine the extent of sample preparation and homogenization necessary for successful PLM or other analysis. Fibers and other components should be removed for further qualitative PLM examination.

Using the information from the stereomicroscopic examination, selection of additional preparation and analytical procedures should be made. Stereomicroscopic examination should typically be performed again after any change or major preparation (ashing, acid dissolution, milling, etc.) to the sample. Stereomicroscopic examination for estimation of asbestos content may also be performed again after the qualitative techniques have clarified the identities of the various fibrous components to assist in resolving differences between the initial quantitative estimates made during the stereomicroscopic analysis and those of subsequent techniques. Calibration of analysts by use of materials of known asbestos content is essential.

The stereomicroscopic examination is often an iterative process. Initial examination and estimates of asbestos concentration should be made. The sample should then be analyzed by PLM and possibly other techniques. These results should be compared to the initial stereomicroscopic results. Where necessary, disagreements between results of the techniques should be resolved by reanalyzing the sample stereomicroscopically.

2.1.6 Calibration Materials

Calibration materials fall into several categories, including internal laboratory standards and other materials that have known asbestos weight percent content. These calibration materials could include:

- Actual bulk samples: asbestos-containing materials that have been characterized by other analytical methods such as XRD, AEM and/or gravimetry. (e.g. NVLAP test samples).

- Generated samples: in-house standards that can be prepared by mixing known quantities of asbestos and known quantities of asbestos-free matrix materials (by weight), and mixing (using blender, mill, etc.) thoroughly to achieve homogeneity; matrix materials such as vermiculite, perlite, sand, fiberglass, calcium carbonate, etc. may be used. A range of asbestos concentrations should be prepared (e.g. 1, 3, 5, 10, 20%, etc.). The relationship between specific gravities of the components used in standards should be considered so that weight/volume relationships may be determined.

- Photographs, drawings: photomicrographs of standards, computer-generated drawings, etc.

Suggested techniques for the preparation and use of in-house calibration standards are presented in Appendix C, and at greater length by Harvey et al.[1] The use of synthesized standards for analyst calibration and internal laboratory quality control is not new however, having been outlined by Webber et al.[2] in 1982.

2.1.7 References

1. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991, pp. 22-29.

2. Webber, J. S., A. Pupons and J. M. Fleser, "Quality-Control Testing for Asbestos Analysis with Synthetic Bulk Materials". **American Industrial Hygiene Associations Journal**, 43, 1982, pp. 427-431.

2.2 Polarized Light Microscopy

2.2.1 Principle and Applicability

Samples of bulk building materials taken for asbestos identification should first be examined with the simple stereomicroscope to determine homogeneity and preliminary fiber identification. Subsamples should then be examined using PLM to determine optical properties of constituents and to provide positive identification of suspect fibers.

The principles of optical mineralogy are well-established.[1,2,3,4] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows for the determination of refractive indices relative to specific crystallographic orientations. Morphology and color are also observed while viewing under plane polarized light. Observation of particles or fibers while oriented between polarizing filters whose privileged vibration directions are perpendicular (crossed polars) allows for determination of isotropism/anisotropism, extinction characteristics of anisotropic particles, and calculation of birefringence. A retardation plate may be placed in the polarized light path for verification of the sign of elongation. If subsamples are prepared in such a way as to represent all sample components and not just suspect fibers, semi-quantitative analysis may also be performed. Semi-quantitative analysis involves the use of calibrated visual area estimation and/or point counting. Visual area estimation is a semi-quantitative method that must relate back to calibration materials. Point counting, also semi-quantitative, is a standard technique used in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[3] and the interpretation of point count data[5] is available.

Although PLM analysis is the primary technique used for asbestos determination, it can show significant bias leading to false negatives and false positives for certain types of materials. PLM is limited by the visibility of the asbestos fibers. In some samples the fibers may be reduced to a diameter so small or masked by coatings to such an extent that they cannot be reliably observed or identified using PLM.

2.2.2 Range

The detection limit for visual estimation is a function of the quantity of sample analyzed, the nature of matrix interference, sample preparation, and fiber size and distribution. Asbestos may be detected in concentrations of less than one percent by area if sufficient material is analyzed. Since floor tiles may contain fibers too small to be resolved by PLM ($< 0.25\ \mu m$ in diameter), detection of those fibers by this method may not be possible. When point counting is used, the detection limit is directly proportional to the amount of sample analyzed, but is also limited by fiber visibility. Quantitation by area estimation, both visual and by point counting, should yield similar results if based on calibration standards.

2.2.3 Interferences

Fibrous and nonfibrous, organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Binder/matrix materials may coat fibers, affect color, or obscure optical characteristics to the extent of masking fiber identity. Many organic mastics are soluble in refractive index liquids and, unless removed prior to PLM examination, may affect the refractive index measurement of constituent materials. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Gravimetric procedures for the removal of interfering materials are presented in Section 2.3.

2.2.4 Precision and Accuracy

Data obtained for samples containing a single asbestos type in a sample matrix have been reported previously by Brantley et al.[6] Data for establishing the accuracy and precision of the method for samples with various matrices have recently become available. Perkins,[7] Webber et al.[8] and Harvey et al.[9] have each documented the tendency for visual estimates to be high when compared to point-count data. Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonably expanded errors, such as those shown in Table 2-1, should be generated for different concentrations of asbestos.

If the laboratory cannot demonstrate adequate precision and accuracy (documented by control charts, etc), quantitation by additional methods, such as gravimetry, may be required. Refer to the Handbook for SRM Users[10] for additional information concerning the concepts of precision and accuracy.

TABLE 2-1. SUGGESTED ACCEPTABLE ERRORS FOR PLM ANALYSIS
(Based on 400 point counts of a reasonably homogeneous sample or 100 fields of view for visual estimate)

% Area Asbestos	Acceptable Mean Result	% Area Asbestos	Acceptable Mean Result
1	>0-3%	50	40-60%
5	>1-9%	60	50-70%
10	5-15%	70	60-80%
20	10-30%	80	70-90%
30	20-40%	90	80-100%
40	30-50%	100	90-100%

2.2.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations must be carried out in a HEPA-filtered, a class 1 biohazard hood, or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.2.5.1 Sample Preparation

Slide mounts are prepared for the identification and quantitation of asbestos in the sample.

2.2.5.1.1 Qualitative Analysis Preparation

The qualitative preparation must allow the PLM analysis to classify the fibrous components of the sample as asbestos or nonasbestos. The major goal of the qualitative

preparation is to mount easily visible fibers in appropriate refractive index liquids for complete optical characterization. Often this can be accomplished by making immersion grain mounts of random subsamples of the homogeneous material. Immersion liquids with refractive indices close to the suspected (see stereomicroscopic analysis) asbestos mineral should be used for the qualitative analysis so that n_D can be determined. Problem samples include those with inhomogeneities, coatings, small fibers, and interfering compounds. Additional qualitative preparations are often necessary for these types of samples. All samples, but especially those lacking homogeneity, may require picking of fibers from specific sample areas during the stereomicroscopic examination. Coatings on the fibers often need to be removed by mechanical or chemical means. Teasing the particles apart or use of a mortar and pestle or similar mechanical method often is sufficient to free fibers from coatings. Chemical means of removing some coatings and interfering compounds are discussed in Section 2.3, Gravimetry.

2.2.5.1.2 Quantitative Analysis Preparation

The major purpose of the quantitative preparation is to provide the analyst with a representative grain mount of the sample in which the asbestos can be observed and distinguished from the nonasbestos matrix. This is typically performed by using randomly selected subsamples from a homogeneous sample (see stereomicroscopic analysis). Particles should be mounted in a refractive index (RI) liquid that allows the asbestos to be visible and distinguished from nonasbestos components. Care should be taken to ensure proper loading and even distribution of particles. Both the qualitative and quantitative sample preparations are often iterative processes. Initial samples are prepared and analyzed. The PLM analysis may disclose problems or raise questions that can only be resolved by further preparations (e.g. through the use of different RI immersion liquids, elimination of interfering compounds, sample homogenization, etc.)

For layered materials, subsamples should be taken from each individual or discrete layer. Each of these subsamples should be treated as a discrete sample, but as stated in Section 2.1.5.2, the results for the individual layers or materials may be combined if called for by monitoring requirements.

Homogenization involves the use of any of a variety of devices, such as a mortar and pestle, mill, or blender to pulverize, disaggregate and mix heterogeneous, friable bulk materials. Selection of the appropriate device is dependent upon personal preference and the nature of the materials encountered. A blender or mortar and pestle may be adequate for homogenizing materials that lack appreciable amounts of tacky matrix/binder, and for separating interfering components from the fibers. For materials which are unusually sticky or tacky, or contain unusually long asbestos fibers, milling (especially freezer milling) may be more efficient. However, milling should be discontinued as soon as the material being milled appears homogeneous, in order to reduce the potential for mechanically reducing fiber size below the resolving power of the polarizing microscope. Hammer mills or cutting mills may also be used on these materials; however, the same precaution regarding reduction of fiber size should be taken. Blending /milling devices should be disassembled (to the extent possible) and thoroughly cleaned after each use to minimize contamination.

2.2.5.2 Analysis

Analysis of bulk building materials consists of the identification and semi-quantitation of the asbestos type(s) present, along with the identification, where possible, of fibrous nonasbestos materials, mineral components and matrix materials. If the sample is heterogeneous due to the presence of discrete layers or two or more distinct building materials, each layer or distinct material should be analyzed, and results reported. Total asbestos content may also be stated in terms of a relative percentage of the total sample.

2.2.5.2.1 Identification

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology
- Color and, if present, pleochroism
- Refractive indices ($\pm$ .005)
- Birefringence
- Extinction characteristics
- Sign of elongation

Descriptions of the optical properties listed above for asbestos fibers may be found in Appendix A, Glossary of Terms. Table 2-2 lists the above properties for the six types of asbestos and Table 2-3 presents the central stop dispersion staining colors for the asbestos minerals with selected high-dispersion index liquids. Tables 2-4 and 2-5 list selected optical properties of several mineral and man-made fibers. All fibrous materials in amounts greater than trace should be identified as asbestos or nonasbestos, with all optical properties measured for asbestos and at least one optical property measured for each nonasbestos fibrous component that will distinguish each from asbestos. Small fiber size and/or binder may necessitate viewing the sample at higher magnification (400-500x) than routinely used (100x).

Although it is not the purpose of this section to explain the principles of optical mineralogy, some discussion of the determination of refractive indices is warranted due to its importance to the proper identification of the asbestos minerals. Following is a brief discussion of refractive index determination for the asbestos minerals.

All asbestos minerals are anisotropic, meaning that they exhibit different optical properties (including indices of refraction) in different directions. All asbestos minerals are biaxial, meaning that they have one principal refractive index parallel (or nearly parallel) to the length of the fiber and two principal refractive indices (plus all intermediate indices between these two) in the plane perpendicular (or nearly so) to the length of the fiber. Although chrysotile (serpentine) is classified as a biaxial mineral, it behaves as a uniaxial mineral (two principal refractive indices) due to its scrolled structure. Amosite and crocidolite, although also biaxial, exhibit uniaxial properties due to twinning of the crystal structure and/or random orientation of fibrils in a bundle around the long axis of the bundle. For all of the asbestos minerals except crocidolite, the highest refractive index (γ) is aligned with the fiber length (positive sign of elongation). For crocidolite, the lowest refractive index (α) is aligned with the fiber length (negative sign of elongation). A more complete explanation of the relationship of refractive indices to the crystallographic directions of the asbestos minerals may be found in References 1, 2, 4, 11 and 12. It should be noted that for the measurement of refractive indices in an anisotropic particle (e.g. asbestos fibers), the orientation of the particle is quite critical. Orientation with respect to rotation about the axis

of the microscope (and thus with respect to the vibration directions of the polarizer and analyzer) and also to the horizontal plane (plane of the microscope stage) will affect the determination of the correct values for refractive indices. The refractive index that is measured will always correspond to a direction perpendicular to the axis of the microscope (i.e., lying in the plane of the stage) and is the direction in that horizontal plane parallel to the vibration direction of the polarizer, by convention E-W.

To determine $\gamma(n \parallel)$ for chrysotile, anthophyllite and amosite, the index is measured when the length of the fiber is aligned parallel to the vibration direction of the polarizer (E-W). Under crossed polars, the fiber should be at extinction in this orientation. To determine the lowest refractive index, α ($n \perp$), for chrysotile and amosite, the fiber should be oriented N-S (extinction position under crossed polars). The determination of $n \parallel$ and $n \perp$ with crocidolite is accomplished in the same manner as with amosite and chrysotile with the exception that the α and γ directions are reversed. For crocidolite, α is measured at the E-W position (parallel to the polarizer) and γ is measured at the N-S orientation (perpendicular to the polarizer). For anthophyllite, the fiber should be oriented N-S and the lowest and highest indices for this orientation should be measured. These correspond to α and β respectively.

The extinction behavior of tremolite-actinolite is anomalous compared to that of most monoclinic minerals due to the orientation of the optic axes relative to the crystallographic axes. This relationship is such that the refractive indices of the principal axes α and γ <u>are not measured</u> when the fiber is exhibiting the maximum extinction angle. The values measured at these positions are α' and γ' The fiber exhibits an extinction angle within a few degrees of the maximum throughout most of its rotation. A wide range of refractive indices from α' to α, and from γ' to γ, are observed. For tremolite-actinolite, β is measured on those fibers displaying parallel extinction when oriented in the N-S position. The refractive index for α is also measured when the fiber is oriented generally in the N-S position and exhibits <u>the true extinction</u> angle; true α will be the <u>minimum</u> index. To determine the refractive index for γ, the fibers should be oriented E-W and exhibit the true extinction angle; true γ will be the <u>maximum</u> value for this orientation.

When viewing single fibers, the analyst may often be able to manipulate the microscope slide cover slip and "roll" the fibers to positions that facilitate measuring the true values of refractive indices. When viewing a large population of fibers with the microscope in the dispersion staining mode, the analyst can easily detect fibers that exhibit the highest and lowest indices (β and α) in the N-S position and the highest indices (γ) in the E-W position. Since individual asbestos fibrils cannot generally be resolved using polarized light microscopy, refractive indices are most commonly measured on fiber bundles. Such measurements would not result in true values for the indices and therefore by convention should be reported as α' and γ'.

Asbestos types chrysotile, amosite and crocidolite are currently available as SRM 1866 and actinolite, tremolite and anthophyllite as SRM 1867 from the Office of Standard Reference Materials, National Institute of Standards and Technology.

2.2.5.2.2 Quantitation of Asbestos Content

As described in Sections 2.1.5 and 2.1.6, a calibrated visual volume estimation of the relative concentrations of asbestos and nonasbestos components should be made during the stereomicroscopic examination. In addition, quantitation of asbestos content should be performed on subsample slide mounts using calibrated visual area estimates and/or a point counting procedure. Section 2.1.6 and Appendix C discuss the procedures for preparation and use of calibration standards. After thorough PLM analysis in which the asbestos and other components of the bulk material are identified, several slides should be carefully prepared from randomly selected subsamples. If the sample is not homogeneous, some homogenization procedure should be performed to ensure that slide preparations made from small pinch samples are representative of the total sample. Homogenization may range from gentle mixing using a mortar and pestle to a brief period of mixing using a blender equipped with a mini-sample container. The homogenization should be of short duration ($\sim$ 15 seconds) if using the blender technique so as to preclude a significant reduction in fiber size. The use of large cover slips (22x30mm) allows for large subsamples to be analyzed. Each slide should be checked to ensure that the subsample is representative, uniformly dispersed, and loaded in a way so as not to be dominated by superimposed (overlapping) particles.

During the qualitative analysis of the sample, the analyst should decide on the appropriate optical system (including magnification) to maximize the visibility of the asbestos in the sample while still allowing the asbestos to be uniquely distinguished from the matrix materials. The analyst may choose to alter the mounting medium or the optical system to enhance contrast. During the quantitative analysis, slides should be scanned using an optical setup that yields the best visibility of the asbestos. Upon finding asbestos, the parameters that were selected in the qualitative analysis for uniquely distinguishing it from the matrix should be used for identification. These properties will vary with the sample but include any or all of the parameters required for the qualitative analysis. For instance, low magnification allows for concurrent use of dispersion staining (focal screening), but compromises resolution of extremely small diameter fibers; use of a compensator plate and crossed polarizers frequently enhances the contrast between asbestos fibers and matrix material.

Visual area estimates should be made by comparison of the sample to calibration materials that have similar textures and fiber abundance (see Section 2.1.6 and Appendix C). A minimum of three slide mounts should be examined to determine the asbestos content by visual area estimation. Each slide should be scanned in its entirety and the relative proportions of asbestos and nonasbestos noted. It is suggested that the ratio of asbestos to nonasbestos material be recorded for several fields for each slide and the results be compared to data derived from the analysis of calibration materials having similar textures and asbestos content.

For point counting, an ocular reticle (cross-line or point array) should be used to visually superimpose a point or points on the microscope field of view. The cross-line reticle is preferred. Its use requires the scanning of most, if not all, of the slide area, thereby minimizing bias that might result from lack of homogeneity in the slide preparation. In conjunction with this reticle, a click-stop counting stage can be used to preclude introducing bias during slide advancement. Magnification used will be dictated by fiber visibility. The slide should be examined along multiple parallel traverses that adequately cover the sample area. The analyst should score (count) only points directly over occupied (nonempty) areas. Empty points should not be scored on the basis of the closest particle. If an asbestos fiber and a nonasbestos particle overlap so that a point is superimposed on their visual intersection,

a point should be scored for both categories. If the point(s) is/are superimposed on an area which has several overlapping particles, the slide should be moved to another field. While not including them in the total asbestos points counted, the analyst should record the presence of any asbestos detected but not lying under the reticle cross-line or array points. A minimum of 400 counts (maximum of eight slides with 50 counts each to minimum of two slides with 200 counts each) per sample is suggested, but it should be noted that accuracy and precision improve with number of counts. Point counting provides a determination of the projected area percent asbestos. Conversion of area percent to dry weight percent is not feasible unless the specific gravities and relative volumes of the different materials are known. It should be noted that the total amount of material to be analyzed is dependent on the asbestos concentration, i.e. the lower the concentration of asbestos, the larger the amount of sample that should be analyzed, in both the visual estimation and point counting methods. Quantitation by either method is made more difficult by low asbestos concentration, small fiber size, and presence of interfering materials.

It is suggested that asbestos concentration be reported as volume percent, weight percent or area percent depending on the method of quantitation used. A weight concentration cannot be determined without knowing the relative specific gravities and volumes of the sample components.

Mineral	Morphology and Color[1]	Refractive Indices[2] α	Refractive Indices[2] γ[5]	Birefringence[6]	Extinction	Sign of Elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless[3]	1.493-1.546 1.532-1.549 1.529-1.559 1.544-1.553	1.517-1.557 1.545-1.556 1.537-1.567 1.552-1.561	0.004-0.017	Parallel	+ (length slow)
Amosite (asbestiform grunerite)	Straight to curved, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so.[4] Opaque inclusions may be present	1.657-1.663 1.663-1.686 1.663-1.686 1.676-1.683	1.699-1.717 1.696-1.729 1.696-1.729 1.697-1.704	0.021-0.054	Usually parallel	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight to curved, rigid fibers. Aspect ratio typically > 10:1. Thick fibers and bundles common, blue to dark-blue in color. Pleochroic.	1.693 1.654-1.701 1.680-1.698	1.697 1.668-1.717 1.685-1.706	0.003-0.022	Usually parallel	- (length fast)
Anthophyllite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Anthophyllite cleavage fragments may be present with aspect ratios <10:1. Colorless to light brown.	1.598-1.652 1.596-1.694 1.598-1.674 1.6148[7]	1.623-1.676 1.615-1.722 1.615-1.697 1.6362[7]	0.013-0.028	Parallel	+ (length slow)
Tremolite-Actinolite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Cleavage fragments may be present with aspect ratios <10:1. Colorless to pale green	Tremolite 1.600-1.628 1.604-1.612 1.599-1.612 1.6063[7]	 1.625-1.655 1.627-1.635 1.625-1.637 1.6343[7]	 0.017-0.028	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)
		Actinolite 1.600-1.628 1.612-1.668 1.613-1.628 1.6126[7]	 1.625-1.655 1.635-1.688 1.638-1.655 1.6393[7]	 0.017-0.028		

[1]Colors cited are seen by observation with plane polarized light.

[2]From references 2, 11, 12, and 18, respectively. Refractive indices for n_d at 589.3nm.

[3]Fibers subjected to heating may be brownish. (references 13, 14, and 15)

[4]Fibers subjected to heating may be dark brown and pleochroic. (references 13, 14, and 15)

[5]∥ to fiber length, except ⊥ to fiber length for crocidolite only.

[6]Maximum and minimum values from references 2, 11, 12, and 18 given.

[7]± 0.0007

TABLE 2-3. TYPICAL CENTRAL STOP DISPERSION STAINING COLORS[1]

Mineral	Cargille® RI Liquid	n∥	n⊥
Chrysotile	1.550HD	Magenta to light blue-green λ_0's ca. 520-620nm	Blue-green to pale blue λ_0's ca. 600-700nm
Amosite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Blue magenta to light blue λ_0's ca. 560-660nm
Crocidolite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
Anthophyllite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Tremolite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Actinolite-asbestos	1.605HD	Pale yellow λ_0's ca. 260-360nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
	1.630HD	Yellow to magenta λ_0's ca. 420-520nm	Golden yellow to blue λ_0's ca. 450-600nm

[1]Modified from reference 16

TABLE 2-4. OPTICAL PROPERTIES OF MAN-MADE TEXTILE FIBERS[1,2]

Fiber Type	n∥	n⊥	n∥ − n⊥	Sign of Elongation
Polyester (Dacron®)	1.710	1.535	0.175	+
Polyamide (Nylon®)	1.582	1.514	0.063	+
Aramid (Kevlar®)	≈2.37	≈1.641	0.729	+
Olefin (Polyethylene)	1.556	1.512	0.044	+
Olefin (Polypropylene)	1.520	1.495	0.025	+
Viscose Rayon	1.535-1.555	1.515-1.535	0.020	+
Acetate	1.478-1.480	1.473-1.476	0.004-0.005	+
Acrylic (Orlon®)	1.505-1.515	1.507-1.517	0.004-0.002	
Modacrylic (Dynel®)	1.535	1.532	0.002	+

[1]Modified from reference 17

[2]Refractive indices for specific fibers; other fibers may vary

TABLE 2-5. OPTICAL PROPERTIES OF SELECTED FIBERS[1]

FIBER TYPE	MORPHOLOGY	REFRACTIVE INDICES	BIREFRINGENCE ($n\parallel - n\perp$)	EXTINCTION ANGLE	SIGN OF ELONGATION	DISPERSION STAINING COLORS
Paper (Cellulose)	Tapered, flat ribbons	$n\parallel$ ~ 1.580 $n\perp$ ~ 1.530	High (0.05)	Parallel and incomplete	+	in 1.550HD $n\parallel$: yellow (λ_o's < 400nm) $n\perp$: pale blue (λ_o's > 700nm)
Olefin (polyethylene)	Filaments or shredded like chrysotile	$n\parallel$ ~ 1.556 $n\perp$ ~ 1.512	Moderate (0.044)	Parallel	+	in 1.550HD $n\parallel$: yellow to magenta (λ_o's = 440-540nm) $n\perp$: pale blue (λ_o's > 700nm)
Brucite (nemalite)	Straight fibers	$n\parallel$ ~ 1.560-1.590 $n\perp$ ~ 1.580-1.600	Moderate (0.012-0.020)	Usually parallel	- occasionally +	in 1.550HD $n\parallel$: golden yellow (λ_o's 440-460nm) $n\perp$: yellow (λ_o's 400-440nm)
Heated amosite	Similar to unheated, (brittle and shorter) pleochroic: $n\parallel$-dark brown $n\perp$ yellow	$n\parallel$ and $n\perp$ >1.700[2]	High (> 0.05)	Usually parallel	+	in 1.680HD $n\parallel$ & $n\perp$: both pale yellow to white (λ_o's < 400nm)
Glass fibers, Mineral wool	Exotic shapes, tear drops, single filaments	1.515-1.700	Isotropic	------	-	in 1.550HD usually pale blue to blue (λ_o's 580 to > 700nm)
Wollastonite	Straight needles and blades	$n\parallel$ ~ 1.630 $n\perp$ ~ 1.632 $n\perp$ also 1.610	Moderate to low (0.018 to 0.002)	Parallel and oblique	+ and -	in 1.605HD $n\parallel$ & $n\perp$, yellow to pale yellow (λ_o's < 460nm)
Fibrous talc	Thin cleavage ribbons and wavy fibers	$n\parallel$ ~ 1.60 $n\perp$ ~ 1.54	High (0.06)	Parallel and oblique	+	in 1.550HD $n\parallel$: pale yellow (λ_o's <400nm) $n\perp$: pale blue (λ_o's >660nm)

[1]From reference 19

[2]From references 13, 14, and 15

2.2.5.2.3 Microscope Alignment

In order to accurately measure the required optical properties, a properly aligned polarized light microscope must be utilized. The microscope is aligned when:

1) the privileged directions of the substage polarizer and the analyzer are at 90° to one another and are represented by the ocular cross-lines;

2) the compensator plate's privileged vibration directions are 45° to the privileged directions of the polarizer and analyzer;

3) the objectives are centered with respect to stage rotation; and,

4) the substage condenser and iris diaphragm are centered in the optic axis.

Additionally, the accurate measurement of the refractive index of a substance requires the use of calibrated refractive index liquids. These liquids should be calibrated regularly to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or R.I. glass beads.

2.2.6 References

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16. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.

17. McCrone, Walter C., "Routine Detection and Identification of Asbestos", **The Microscope**, 33, 1985, pp. 273-284

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2.3 Gravimetry

2.3.1 Principle and Applicability

Many components of bulk building materials, specifically binder components, can be selectively removed using appropriate solvents or, in the case of some organics, by ashing. The removal of these components serves the following purposes:

1) to isolate asbestos from the sample, allowing its weight to be determined;

2) to concentrate asbestos and therefore lower the detection limit in the total sample;

3) to aid in the detection and identification of fibrous components; and,

4) to remove organic (ashable) fibers which are optically similar to asbestos.

Common binder materials which are removed easily using the techniques described include: 1) calcite, gypsum, magnesite, brucite, bassanite, portlandite, and dolomite, using hydrochloric acid, and 2) vinyl, cellulose, and other organic components, by ashing. The removal of the binder components results in a residue containing asbestos, if initially present, and any other non-soluble or non-ashable components which were present in the original sample. Unless the procedures employed result in the loss of some asbestos, the weight percent of the residue is the upper limit for the weight percent of asbestos in the sample.

This section describes the procedure for removing acid-soluble and ashable components, and for determining the weight percent of the residue. However, the acid dissolution and ashing techniques can be used without the accompanying weight measurements to either liberate or clean fibers to aid in qualitative PLM or AEM analyses.

This technique is not an identification technique. Other methods, such as PLM, XRD, or AEM must be used to determine the identity of the components. A description of the suggested apparatus, reagents, etc. needed for the techniques described is included in Appendix B.

2.3.2 Interferences

Any components which cannot by removed from the sample by selective dissolution or ashing interfere with asbestos quantitation. These components include, but are not limited to, many silicates (micas, glass fibers, etc.) and oxides (TiO_2, magnetite, etc.). When interfering phases are present (the residue contains other phases in addition to asbestos), other techniques such as PLM, AEM, or XRD must be used to determine the percent of asbestos in the residue.

Care must be taken to prevent loss of or chemical/structural changes in the critical components (asbestos). Prolonged exposure to acids or excessive heating (above 500°C) can cause changes in the asbestos components in the sample and affect the optical properties.[1,2,3]

2.3.3 Quantitation

The weight of the residue remaining after solvent dissolution/ashing should be compared with the original weight of the material. Presuming no insoluble material is lost, the weight percent of the residue is the upper limit for the amount of asbestos in the sample. If the residue is comprised only of asbestos, then the weight percent of residue equals the weight percent of asbestos in the sample. If the residue contains other phases, then techniques such as PLM, XRD, or AEM must be employed to determine the relative abundance of asbestos in the residue.

The precision and accuracy of the technique are dependent upon the homogeneity of the material, the accuracy of the weight measurements, and the effectiveness of the sample reduction and filtering procedures. In practice, the precision can be equal to $\pm 1\%$, and the accuracy at 1 wt% asbestos can be less than or equal to $\pm 10\%$ relative.

The incomplete solution of components and the presence of other nonasbestos components in the residue contribute to producing a positive bias for the technique (falsely high percentages of asbestos).

2.3.4 Preliminary Examination and Evaluation

Stereomicroscopic and PLM examinations of the sample should already have been conducted prior to initiating this procedure. These examinations should have provided information about: 1) whether the sample contains components which can be removed by acid-washing, solvent dissolution, or ashing, and 2) whether the sample contains asbestos, or fibers that might be asbestos, or whether no asbestos was detected.

If the sample is friable and contains organic (ashable) components, the ashing procedure should be followed. If the sample is friable and contains HCl-soluble components, the acid dissolution procedure should be followed. If the sample is friable and contains both types of

components, the two procedures can be applied, preferably with acid dissolution following ashing.

If the sample is nonfriable (e.g. floor tiles), it is also recommended that the ashing procedure be used first, followed by the acid dissolution procedure. The ashing procedure reduces floor tiles to a material which is easily powdered, simplifying the sample preparation for acid dissolution.

2.3.5 Sample Preparation

2.3.5.1 Drying

Any moisture in the sample will affect the weight measurements, producing falsely low percentages of residue. If the sample is obviously wet, it should be dried at low temperature (using a heat lamp, or simply by exposure at ambient conditions, prior to starting the weighing procedure). If an oven is used, the drying temperature should not exceed 60°C. Drying by means of heat lamp or ambient air must be performed within a safety-filtered hood. Even if the sample appears dry, it can contain enough moisture to affect the precision and accuracy of the technique. The test for sample moisture involves placing the amount of sample to be used on the weighing pan; if the weight remains stable with time, then the sample is dry enough. If the weight decreases as the sample sits on the weighing pan, then the sample should be dried. Where conditions of moderate to high humidity are known to exist, all materials to be weighed should be allowed time to stabilize to these ambient conditions.

2.3.5.2 Homogenization/Grain Size Reduction

To increase the accuracy and precision of the acid dissolution technique, the sample should be homogenized prior to analysis. This reduces the grain size of the binder material and releases it from fiber bundles so that it may be dissolved in a shorter time period. Leaving the sample in the acid for a longer period of time to complete the dissolution process can adversely affect the asbestos components, and is not recommended. Homogenization of the sample also ensures that any material removed for analysis will more likely be representative of the entire sample.

Homogenization of friable samples prior to ashing may also accelerate the ashing process; however, the ashing time can simply be increased without affecting the asbestos in the sample. Nonfriable samples, such as vinyl floor tiles, can be broken or shaved into pieces to increase surface area and accelerate the ashing process.

Homogenization and grain size reduction can be accomplished in a variety of ways: 1) hand grinding in a mortar and pestle; 2) crushing with pliers or similar instrument; 3) mixing in a blender; 4) milling (i.e. Wylie mill, cryomill, etc.); or 5) any other technique which seems suitable. If the fibers are extremely long, a pair of scissors or similar implement can be used to reduce the fiber length.

2.3.6 Procedure for Ashing

1) **Weigh appropriate amount of material.**

   There is no restriction on the maximum weight of material used; however, a large amount of material may take longer to ash. Enough material should be used to avoid a significant contribution of weighing errors to the total accuracy and precision.

2) **Place material in crucible, weigh, and cover with lid.**

   Placing a lid on the crucible both minimizes the amount of oxygen available, slowing the rate of combustion of the sample, and prevents any foreign material from falling into the crucible during ashing.

3) **Place crucible into furnace, and ash for at least 6 hours.**

   The furnace temperature at the sample position should be at least 300°C but should not exceed 500°C. If the sample combusts (burns), the temperature of the sample may exceed 500°C. Chrysotile will decompose above approximately 500°C.

   The furnace area should be well-ventilated and the fumes produced by ashing should be exhausted outside the building.

   The ashing time is dependent on the furnace temperature, the amount of sample, and the surface area (grain size). Six hours at 450°C is usually sufficient.

4) **Remove crucible from furnace, allow contents to adjust to room temperature and humidity, and weigh.**

5) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

6) **Analyze residue and/or proceed to acid dissolution procedure.**

If the objective was to remove organic fibers that may be confused optically with asbestos, examine residue with PLM to determine whether any fibers remain.

If the sample is a floor tile, the acid dissolution procedure must now be performed. The residue does not have to be analyzed at this stage.

2.3.7 Use of Solvents for Removal of Organics

Solvent dissolution may be used as a substitute for low temperature ashing for the purpose of removing organic interferences from bulk building materials. However, solvent dissolution, because of the involvement of potentially hazardous reagents such as tetrahydrofuran, amyl acetate, 1-1-1, trichlorethane, etc., requires **that all work be performed with extreme caution inside a biohazard hood**. Material Safety Data Sheets should be reviewed before using any solvent. Solvent dissolution involves more apparatus than does ashing, and requires more time, mainly due to set-up and slow filtration resulting from viscous solvent/residue mixtures.

The following is a brief description of the solvent dissolution process.

1) **Weigh starting material.**

Place approximately 15-25ml of solvent in a 100ml beaker. Add 2.5-3.0 grams (carefully weighed for continued gravimetric tracking) of powdered sample.

2) **Untrasonicate sample.**

Place the beaker in an ultrasonic bath (or ultrasonic stirrer) for approximately 0.5 hours. The sample containers should be covered to preclude escape of an aerosol spray.

3) **Centrifuge sample.**

Weigh centrifuge vial before adding beaker ingredients. Wash beaker with an additional 10-15ml of solvent to remove any remaining concentrate. Then centrifuge

at approximately 2000-2500 rpm for 0.5 hour. Use solvent-resistant centrifuge tubes.

4) **Decant sample, reweigh.**

After separation by centrifuging, decant solvent by pipetting. Leave a small amount of solvent in the centrifuge vial to minimize the risk of decanting solid concentrate. Allow solid concentrate to dry in vial, then reweigh.

2.3.8 Procedure for Acid Dissolution

1) **Weigh starting material, transfer to acid resistant container.**

Small, dry sample weights between 0.1g and 0.5g are recommended (determined for 47mm filters adjust amount if different diameter filters are used). If too much material is left after acid dissolution the filter can get clogged and prevent complete filtration. Very small samples are also to be avoided, as the weighing errors will have a large effect on the total accuracy and precision of the technique.

2) **Weigh filter.**

3) **Add HCl to sample in container, stir, allow to sit for 2-10 minutes.**

Either concentrated or dilute HCl can be used. If concentrated HCl is used, add enough acid to completely soak the material, allow the reaction to proceed to completion, and then dilute with distilled water. Alternatively, a dilute solution, made by adding concentrated HCl to distilled water, can be used in the place of concentrated HCl. A solution of 1 part concentrated HCl to 3 parts distilled water (approximately 3N solution) has been found to be quite effective in removing components within 5 minutes. For a sample size less than 0.5g, 20-30 ml of a 3N HCl solution is appropriate. In either case (using concentrated or dilute HCl), the reaction will be more effective if the sample has been homogenized first. All obvious signs of reaction (bubbling) should cease before the sample is filtered. Add fresh acid, a ml or two at a time, to ensure complete reaction. It should be noted that if dolomite is present, a 15-20 minute exposure to concentrated HCl may be required to completely dissolve the carbonate materials.

NOTE: Other solvents may be useful for selective dissolution of nonasbestos components. For example, acetic acid will dissolve calcite, and will not dissolve asbestos minerals. If any solvent other than hydrochloric acid is used for the dissolution of inorganic components, the laboratory must be able to demonstrate that the solvent does not remove asbestos from the sample.

4) **Filter solution.**

Use the pre-weighed filter. Pour the solution into the vacuum filter assembly, then rinse all material from container into filter assembly. Rinse down the inside walls of the glass filter basin and check for particles clinging to the basin after removal.

5) **Weigh dried filter + residue, subtract weight of filter from total.**

6) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

7) **Analyze residue.**

Perform stereomicroscopic examination of residue (can be performed without removing the residue from the filter). Note in particular whether any binder material is still present.

Perform PLM, AEM, or XRD analysis of residue to identify fibers and determine concentration as described in the appropriate sections of this method.

8) **Modify procedure if necessary.**

If removal of the acid soluble components was not complete, start with a new subsample of material and try any of the following:

a) Decrease grain size of material (by grinding, milling, etc.)
b) Put solutions on hot plate warm slightly
c) Increase soak time (exercise caution)

9) **Calculate relative weight% asbestos in sample.**

wt% asbestos in sample = % asbestos in residue x wt% residue ÷ 100

For floor tiles, if the ashing procedure was used first, multiply the weight % of asbestos in the sample, as determined above, by the weight percent of the residue from the ashing procedure, then divide by 100.

Example:
A = wt% residue from ashing = 70%
B = wt% residue from HCl = 20%
C = wt% of asbestos in HCl residue = 50%

wt% asbestos after HCl dissolution = B x C ÷ 100 = 20 x 50 ÷ 100 = 10%

wt% asbestos in floor tile = (B x C ÷ 100) x A ÷ 100 = 10 x 70 ÷ 100 = 7%

If weights are expressed in decimal form, multiply the weight % of asbestos in the sample by the weight % of the residue from the ashing procedure, then multiply by 100.

wt% asbestos after HCl dissolution = B x C = 0.2 x 0.5 = 0.1 (x 100 = 10%)

wt% asbestos in floor tile = (B x C) x A = 0.1 x 0.7 = 0.07 (x 100 = 7%)

2.3.9 Determination of Optimal Precision and Accuracy

The precision of the technique can be determined by extracting multiple subsamples from the original sample and applying the same procedure to each. The optimal accuracy of the technique can be determined by applying gravimetric standards. Mixtures of calcite and asbestos (chrysotile, amosite, etc.) in the following proportions are recommended for testing the accuracy of the acid dissolution technique: 0.1 wt% asbestos/99.9 wt% calcite, 1.0 wt% asbestos/99.0 wt% calcite, and 10 wt% asbestos/90 wt% calcite. Mixtures of cellulose and asbestos are useful for testing the accuracy of the ashing technique.

Mixtures of only two components, as described above, are simplifications of "real-world" samples. The accuracy determined by analyzing these mixtures is considered optimal and may not apply directly to the measurement of each unknown sample. However, analyzing replicates and standards using the full laboratory procedure, including homogenization, ashing, acid dissolution, filtration, and weighing, may uncover steps that introduce significant bias or variation that the laboratory may then correct.

2.3.10 References

1. Kressler, J. R., "Changes in Optical Properties of Chrysotile During Acid Leaching", **The Microscope**, 31, 1983, pp. 165-172.

2. Prentice, J. and M. Keech, "Alteration of Asbestos with Heat", **Microscopy and Analysis,** March 1989.

3. Laughlin, G. and W. C. McCrone, "The Effect of Heat on the Microscopical Properties of Asbestos", **The Microscope,** 37, 1989, pp. 8-15.

2.4 X-Ray Powder Diffraction

2.4.1 Principle and Applicability

The principle of x-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid crystalline material will diffract an incident beam of parallel, monochromatic x-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the x-ray wavelength, Å;
d = the interplanar spacings of the set of reflecting lattice planes, Å and
θ = the angle of incidence between the x-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident x-ray beam, a diffraction pattern can be generated that will be uniquely characteristic of the structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-6). However, when used in conjunction with methods such as PLM or AEM, XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For **qualitative** analysis by XRD methods, samples should initially be scanned over limited diagnostic peak regions for the serpentine ($\sim$7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-7). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals should be submitted to a full (5°-60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns should be compared with standard reference powder

diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Accurate **quantitative** analysis of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis should be selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] can be used in which, subsequent to comminution of the bulk material to $\sim 10\ \mu m$ by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods[7,8] are commonly used for **semi-quantitative analysis.**

TABLE 2-6. THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform	Chemical Abstract Service No.
Serpentine		
Chrysotile	Antigorite, lizardite	12001-29-5
Amphibole		
Anthophyllite asbestos	Anthophyllite	77536-67-5
Cummingtonite-grunerite asbestos (Amosite)	Cummingtonite-grunerite	12172-73-5
Crocidolite	Riebeckite	12001-28-4
Tremolite asbestos	Tremolite	77536-68-6
Actinolite asbestos	Actinolite	77536-66-4

TABLE 2-7. PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS[1]

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file[2] number
Chrysotile (Serpentine)	7.31_{100} 7.36_{100} 7.10_{100}	3.65_{70} 3.66_{80} 2.33_{80}	4.57_{50} 2.45_{65} 3.55_{70}	21-543[3] 25-645 22-1162 (theoretical)
Amosite (Grunerite)	8.33_{100} 8.22_{100}	3.06_{70} 3.060_{85}	2.756_{70} 3.25_{70}	17-745 (nonfibrous) 27-1170 (UICC)
Anthophyllite	3.05_{100} 3.06_{100}	3.24_{60} 8.33_{70}	8.26_{55} 3.23_{50}	9-455 16-401 (synthetic)
Crocidolite (Riebeckite)	8.35_{100} 8.40_{100}	3.10_{55} 3.12_{55}	2.720_{35} 2.726_{40}	27-1415 (UICC) 19-1061
Actinolite	2.72_{100}	2.54_{100}	3.40_{80}	25-157
Tremolite	8.38_{100} 2.706_{100} 3.13_{100}	3.12_{100} 3.14_{95} 2.706_{60}	2.705_{90} 8.43_{40} 8.44_{40}	13-437[3] 20-1310[3] (synthetic) 23-666 (synthetic mixture w/richterite)

1. This information is intended as a guide only. Complete powder diffraction data, including mineral type and source, should be referred to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite and chrysotile are available from the U.S. Bureau of Mines, Reference 4.

2. From Reference 3

3. Fibrosity questionable

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.4.2 Range and Sensitivity

The range and sensitivity of the method have not been determined. They will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected and their relative intensities, preferred orientation, and instrumental limitations. A detection limit of one percent is feasible given certain sample characteristics.

2.4.3 Limitations

2.4.3.1 Interferences

Since the asbestiform and nonasbestiform analogs of the serpentine and amphibole minerals (Table 2-7) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally-occurring materials, the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials, the nature of the interferences may vary greatly (Table 2-8) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-9 and include the following:

- **Chlorite** has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.31 Å) and secondary (3.65 Å) peaks for serpentine (chrysotile). Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.

- **Vermiculite** has secondary peaks at 7.14 Å and 3.56 Å that could interfere with the primary peak (7.31 Å) and a secondary peak (3.65 Å) of serpentine (chrysotile).

TABLE 2-8. COMMON CONSTITUENTS IN BUILDING MATERIAL

(From Ref. 10)

A. Insulation Materials

Chrysotile
Amosite
Crocidolite
*Rock wool
*Slag wool
*Fiber glass
Gypsum ($CaSO_4 \cdot 2H_20$)
Vermiculite (micas)
*Perlite
Clays (kaolin)
*Wood pulp
*Paper fibers (talc, clay carbonate filters)
Calcium silicates (synthetic)
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in "amosite")
Magnesite
*Diatomaceous earth

B. Flooring Materials

Calcite
Dolomite
Titanium Oxide
Quartz
Antigorite
Chrysotile
Anthophyllite
Tremolite
*Organic binders
Talc
Wollastonite

C. Spray Finishes or Paints

Bassanite
Carbonate minerals (calcite, dolomite, vaterite)
Talc
Tremolite
Anthophyllite
Serpentine (including chrysotile)
Amosite
Crocidolite
*Mineral wool
*Rock wool
*Slag wool
*Fiber glass
Clays (kaolin)
Micas
Chlorite
Gypsum
Quartz
*Organic binders and thickeners
Hydromagnesite
Wollastonite
Opaques (chromite, magnetite inclusion in serpentine)
Hematite (inclusions in "amosite")

D. Cementitious Materials

Chrysotile
Amosite
Crocidolite
Micas
Fiber glass
Cellulose
Animal hair
Quartz
Gypsum
Calcite
Dolomite
Calcium silicates

E. Roofing Materials

Chrysotile
Cellulose
Fiber glass
Mineral Wool
Asphalt
Quartz
Talc
Micas

* Amorphous materials--contribute only to overall scattered radiation and increased background radiation.

TABLE 2-9 INTERFERENCES IN XRD ANALYSIS OF ASBESTIFORM MINERALS

Asbestiform Mineral	Primary diagnostic peaks (approximate d spacings in Å)	Interference
Serpentine Chrysotile	7.3	Nonasbestiform serpentines, (antigorite, lizardite), chlorite, vermiculite, sepiolite, kaolinite, gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite), chlorite, vermiculite, halloysite, cellulose
Amphibole Amosite (Grunerite) Anthophyllite Crocidolite (Riebeckite)	3.1	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences, talc, carbonates
Tremolite Actinolite	8.3	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences

- **Sepiolite** produces a peak at 7.47 Å which could interfere with the primary peak (7.31 Å) of serpentine (chrysotile).

- **Halloysite** has a peak at 3.63 Å that interferes with the secondary (3.65 Å) peak for serpentine (chrysotile).

- **Kaolinite** has a major peak at 7 15 Å that may interfere with the primary peak of serpentine (chrysotile) at 7.31 Å when present at concentrations of > 10 percent. However, the secondary serpentine (chrysotile) peak at 3.65 Å may be used for quantitation.

- **Gypsum** has a major peak at 7.5 Å that overlaps the 7.31 Å peak of serpentine (chrysotile) when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris (bassanite).

- **Cellulose** has a broad peak that partially overlaps the secondary (3.65 Å) serpentine (chrysotile) peak.[8]

- Overlap of major diagnostic peaks of the amphibole minerals, grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the x-ray port.

- **Carbonates** may also interfere with quantitative analysis of the amphibole minerals grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite-actinolite. Calcium carbonate ($CaC0_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, the time in acid should be no more than 20 minutes to preclude any loss of chrysotile.[11]

- A major **talc** peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of actinolite (3.14 Å), riebeckite (crocidolite) (3.10 Å), grunerite (amosite) (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interference is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, resulting in diffraction patterns characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.4.3.2 Matrix Effects

If a copper x-ray source is used, the presence of iron at high concentrations in a sample will result in significant x-ray fluorescence, leading to loss of peak intensity, increased background intensity, and an overall decrease in sensitivity. This situation may be corrected by use of an x-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a

diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5,13,14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7,8] or filter substrate (Ag) peak.[5,6]

2.4.3.3 Particle Size Dependence

Because the intensity of diffracted x-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μm.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.4.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7,8,15] For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.[12,14] However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

2.4.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. The National Institute of Standards and Technology can

provide standard reference materials for chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867).

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be under-estimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4.4 Precision and Accuracy

Neither the precision nor accuracy of this method has been determined. The individual laboratory should obtain or prepare a set of calibration materials containing a range of asbestos weight percent concentrations in combination with a variety of matrix/binder materials. Calibration curves may be constructed for use in semi-quantitative analysis of bulk materials.

2.4.5 Procedure

2.4.5.1 Sampling

Samples taken for analysis of asbestos content should be collected as specified by EPA[19]

2.4.5.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD may be used as an additional technique, both for identification and quantitation of sample components. **Note: Asbestos is a toxic substance. All handling of dry materials should be performed in a safety-hood.**

2.4.5.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin-layer or bulk).

Bulk materials are usually received as heterogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.4.5.3 and 2.4.5.4. **Note: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.**

2.4.5.2.2 Milling

Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[20-23] Therefore, all milling times should be kept to a minimum.

For **qualitative analysis**, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other

suitable wetting agent, to minimize exposure, and the procedure should be performed in a HEPA-filtered hood.

For accurate, reproducible **quantitative analysis**, the particle size of both sample and standard materials should be reduced to $\sim 10\ \mu m$. Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill*, or equivalent) for a maximum time of 10 minutes (some samples may require much shorter milling time) is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to $< 10\ \mu m$.[8,16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.4.5.2.3 Ashing

For materials shown by PLM to contain large amounts of cellulose or other organic materials, it may be desirable to ash prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,[24,25] ashing temperatures should be kept below 500°C. Use of a muffle furnace is recommended. In all cases, calibration of the furnace is essential to ensure that a maximum ashing temperature of 500°C is not exceeded (see Section 2.3).

2.4.5.2.4 Acid Washing

Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.4.3.1), it may be necessary to remove these interferences by a simple acid washing procedure prior to analysis (see Section 2.3).

2.4.5.3 Qualitative Analysis

2.4.5.3.1 Initial Screening of Bulk Material

Qualitative analysis should be performed on a representative, homogeneous portion of the sample, with a minimum of sample treatment, using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.4.5.2.2) to a final particle size sufficiently small ($\sim 100\ \mu m$) to allow adequate packing into a sample holder.

2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. **(Note: Use of back-packing method[26] for bulk sample preparation may reduce preferred orientation effects.)**

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~ 7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-7). The x-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°-60° 2θ; 1° 2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data[3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-7; common constituents of bulk insulation and wall materials are listed in Table 2-8.

2.4.5.3.2 Detection of Minor or Trace Constituents

Routine screening of bulk materials by XRD may fail to detect small concentrations ($< 1\%$) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances when confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.4.5.3.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.4.5.4) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-7). Accurate

transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.4.5.4 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[6] A thick-layer bulk method involving pelletizing the sample may be used for semi-quantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.4.5.2.2.

2. Dry at 60°C for 2 hours; cool in a desiccator.

3. Weigh accurately to the nearest 0.01 mg.

4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.4.5.2.3 and 2.4.5.2.4. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss, L.

5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask containing approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.

6. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the sample material.

7. Dilute to volume with isopropanol.

8. Place flask on a magnetic-stirring plate. Stir.

9. Place silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw

an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and repeat the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has (have) been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-7).

12. Analyze the selected diagnostic reflection(s) by step-scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I°_{Ag} Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net count, I°_r, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I°_r:

$$\hat{I}_a = \frac{I_a}{I_r^\circ}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_r^\circ}, \text{ and } \hat{I}_{Ag}^\circ = \frac{I_{Ag}^\circ}{I_r^\circ}$$

2.4.6 Calibration

2.4.6.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.4.5.2.2. **Equivalent standardized matrix reduction and sizing techniques should be used for both standard and sample materials.**

2. Dry at 100°C for 2 hours; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Transfer each to a 1-L volumetric flask containing approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.4.6.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.4.5.4, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, $\hat{I}^{\circ}_{std}$, as outlined in Section 2.4.5.4, step 14.

2.4.7 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\hat{I}^{\circ}_{std}$, on w. Poor reproducibility ($\pm$15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight-line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\hat{I}^{\circ}_{std}$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\hat{I}_{Ag}$ for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak $\hat{I}^{\circ}_{Ag}$, of the sample filter, calculate the transmittance, T, for each sample as follows:[27,28]

$$T = \frac{\hat{I}_{Ag}}{\hat{I}^{\circ}_{Ag}}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a}$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and

θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a(1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;

W_a = mass of asbestos mineral on filter, in μg;

W_T = total sample weight on filter, in μg;

L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.4.5.4).

2.4.8 References

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2.5 Analytical Electron Microscopy

2.5.1 Applicability

Analytical electron microscopy (AEM) can often be a reliable method for the detection and positive identification of asbestos in some bulk building materials, both friable and nonfriable. The method is particularly applicable to bulk materials that contain a large amount of interfering materials that can be removed by ashing and/or dissolution and contain asbestos fibers that are not resolved by PLM techniques. Many floor tiles and plasters would be included in this type of sample. In combination with suitable specimen preparation techniques, the AEM method can also be used to quantify asbestos concentrations.

2.5.2 Range

The range is dependent on the type of bulk material being analyzed. The upper detection limit is 100%, and the lower detection limit can be as low as 0.0001% depending on the extent to which interfering materials can be separated during the preparation of AEM

specimens, the sophistication of the AEM preparation, and the amount of labor expended on AEM examination.

2.5.3 Interferences

The presence of a large amount of binder/matrix materials associated with fibers can make it difficult to positively identify fibers as asbestos. The portion of the fiber examined by either electron diffraction or energy dispersive x-ray analysis (EDXA) must be free of binder/matrix materials.

2.5.4 Precision and Accuracy

The precision and accuracy of the method have not been determined.

2.5.5 Procedures

The procedures for AEM specimen preparation depend on the data required. In analysis of floor tiles, the weighed residue after removal of the matrix components (see Section 2.3, Gravimetry) is often mostly asbestos, and the task is primarily to identify the fibers. In this situation the proportion of asbestos in the residue can be estimated by AEM and this estimate can be used to refine the gravimetric result. For many floor tiles, the final result is not very sensitive to errors in this estimation because the proportion of asbestos in the residue is very high. For samples in which this is not the case, precise measurements can be made using a quantitative AEM preparation, in which each grid opening of the specimen grid corresponds to a known weight of the original sample or of a concentrate derived from the original sample. Asbestos fibers on these grids are then identified and measured, using a fiber counting protocol which is directed towards a precise determination of mass concentration. This latter procedure is suitable for samples of low asbestos concentration, or for those in which it is not possible to remove a large proportion of the matrix material.

2.5.5.1 AEM Specimen Preparation for Semi-Quantitative Evaluation

The residual material from any ashing or dissolution procedures (see Section 2.3) used (usually trapped on a membrane filter) should be placed in a small volume of ethanol or another solvent such as acetone or isopropyl alcohol, in a disposable beaker, and dispersed

by treatment in an ultrasonic bath. A small volume of this suspension (approximately 3μl) should be pipetted onto the top of a carbon-coated TEM grid. The suspension should be allowed to dry under a heat lamp. The grid is then ready for examination.

Samples that are not conducive to ashing or dissolution may also be prepared in this way for AEM analysis. A few milligrams of the sample may be ground in a mortar and pestle or milled, dispersed in ethanol or another solvent using an ultrasonic bath, and pipetted onto a grid as described previously.

2.5.5.2 AEM Specimen Preparation for Quantitative Evaluation

The objective of this preparation is to obtain a TEM grid on which a known weight of the bulk sample is represented by a known area of the TEM grid. A known weight of the bulk sample, or of the residue after extraction, should be dispersed in a known volume of distilled water. Aliquots of this dispersion should then be filtered through 0.22 μm pore-size MCE or 0.2 μm pore-size PC filters, using filtration techniques as described for analysis of water samples.[1] In order to obtain filters of appropriate particulate loading for AEM analysis, it may be necessary to perform serial dilutions of the initial dispersion. TEM grids should then be prepared from appropriately-loaded filters, using the standard methods.[2]

Determination of the mass concentration of asbestos on the TEM grids requires a different fiber counting protocol than that usually used for determination of numerical fiber concentrations. Initially, the grids should be scanned to determine the dimensions of the largest asbestos fiber or fiber bundle on the specimens. The volume of this fiber or bundle should be calculated. The magnification of the AEM should be set at a value for which the length of this fiber or bundle just fills the fluorescent screen. Asbestos fiber counting should then be continued at this magnification. The count should be terminated when the volume of the initial large fiber or bundle represents less than about 5% of the integrated volume of all asbestos fibers detected. This counting strategy ensures that the fiber counting effort is directed toward those fibers which contribute most to the mass, and permits a precise mass concentration value to be obtained.

2.5.5.2.1 Identification

To document the positive identification of asbestos in a sample, the analyst should record the following physical properties: morphology data, electron diffraction data, EDXA data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos should be recorded.

The purpose of the identification data collected is to prevent or limit false negatives and false positives. This can be accomplished by having a system for measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might analytically interfere with asbestos.

2.5.6 References

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2.6 Other Methodologies

Additional analytical methods (e.g. Scanning Electron Microscopy) may be applicable for some bulk materials. However, the analyst should take care to recognize the limitations of any analytical method chosen. Conventional SEM, for example, cannot detect small diameter fibers ($\sim < 0.2\mu m$), and cannot determine crystal structure. It is, however, very useful for observing surface features in complex particle matrices, and for determining elemental compositions.

3.0 QUALITY CONTROL/QUALITY ASSURANCE OPERATIONS- PLM

A program to routinely assess the quality of the results produced by the PLM laboratory must be developed and implemented. **Quality Control (QC)** is a system of activities whose purpose is to control the quality of the product or service so that it meets the need of the users. This also includes **Quality Assessment**, whose purpose is to provide assurance that the overall quality control is being done effectively. While the essential elements of a quality control system are described in detail elsewhere,[1,2,3,4,5,6] only several of the elements will be discussed here. **Quality Assurance (QA)** is comprised of Quality Control and Quality Assessment and is a system of activities designed to provide assurance that a product or service meets defined standards of quality.

The purpose of the Quality Assurance program is to minimize failures in the analysis of materials prior to submitting the results to the client. Failures in the analysis of asbestos materials include false positives, false negatives, and misidentification of asbestos types. False positives result from identification or quantitation errors. False negatives result from identification, detection, or quantitation errors.

For the stereomicroscopic and PLM techniques, the quality control procedures should characterize the accuracy and precision of both individual analysts and the techniques. Analysts should demonstrate their abilities on calibration materials, and also be checked routinely on the analysis of unknowns by comparison with results of a second analyst. The limitations of the stereomicroscopic and PLM techniques can be determined by using a second analytical technique, such as gravimetry, XRD, or AEM. For example, stereomicroscopic and PLM techniques can fail in the analysis of floor tiles because the asbestos fibers in the sample may be too small to be resolved by light microscopy. An XRD or AEM analysis is not subject to the same limitations, and may indicate the presence of asbestos in the sample.

The accuracy, precision, and detection limits of all analytical techniques described in this method are dependent on the type of sample (matrix components, texture, etc.), on the preparation of the sample (homogeneity, grain size, etc.), and the specifics of the method (number of point counts for PLM, mass of sample for gravimetry, counting time for XRD,

etc.). These should be kept in mind when designing quality control procedures and characterizing performance, and are variables that must be tracked in the quality assurance system.

3.1 General Considerations

3.1.1 Training

Of paramount importance in the successful use of this or any other analytical method is the well-trained analyst. It is highly recommended that the analyst have completed course work in optical mineralogy on the collegiate level. That is not to say that others cannot successfully use this method, but the classification error rate[7] may, in some cases, be directly attributable to level of training. In addition to completed course work in optical mineralogy, specialized course work in PLM and asbestos identification by PLM is desirable. Experience is as important as education. A good laboratory training program can be used in place of course work. Analysts that are in training and not yet fully qualified should have all analyses checked by a qualified analyst before results are released. A QC Plan for asbestos identification would be considered incomplete without a detailed description of the analyst training program, together with detailed records of training for each analyst.

3.1.2 Instrument Calibration and Maintenance

Microscope alignment checks (alignment of the polarizer at 90° with respect to the analyzer, and coincident with the cross-lines, proper orientation of the slow vibration direction of the Red I compensator plate, image of the field diaphragm focussed in the plane of the specimen, centering of the central dispersion staining stop, etc.) should be performed with sufficient frequency to ensure proper operations. Liquids used for refractive index determination and those optionally used for dispersion staining should have periodic refractive index checks using a refractometer or known refractive index solids. These calibrations must be documented.

Microscopes and ancillary equipment should be maintained daily. It is recommended that at least once per year each microscope be thoroughly cleaned and re-aligned by a professional microscope service technician. Adequate inventories of replaceable parts

(illumination lamps, etc.) should be established and maintained. All maintenance must be documented.

3.2 Quality Control of Asbestos Analysis

3.2.1 Qualitative Analysis

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite) using combined stereomicroscopic and PLM techniques. Standards for the six asbestos types listed are available from NIST, and should be used to train analysts in the measurement of optical properties and identification of asbestos. These materials can also be used as identification standards for XRD and AEM.

Identification errors between asbestos types (e.g. reporting amosite when tremolite is present) implies that the analyst cannot properly determine optical properties and is relying on morphology as the identification criteria. This is not acceptable. Each analyst in the lab should prove his or her proficiency in identifying the asbestos types; this can be checked through use of calibration materials (NVLAP proficiency testing materials, materials characterized by an independent technique, and synthesized materials) and by comparing results with another analyst. The identification of all parameters (e.g. refractive indices, birefringence, sign of elongation, etc.) leading to the identification should fall within control limits determined by the laboratory. In addition, a subset of materials should be analyzed using another technique to confirm the analysis.

As discussed earlier, the qualitative analysis is dependent upon matrix and asbestos type and texture. Therefore, the quality assurance system should monitor for samples that are difficult to analyze and develop additional or special steps to ensure accurate characterization of these materials. When an analyst is found to be out of the control limits defined by the laboratory, he or she should undergo additional training and have confirmatory analyses performed on all samples until the problem has been corrected.

3.2.2 Quantitative Analysis

The determination of the amount of asbestos in a sample can be accomplished using the various techniques outlined in this method. The mandatory stereomicroscopic and PLM examinations provide concentrations in terms of volume, area, or weight, depending upon the calibration procedure. Gravimetric and quantitative XRD techniques result in concentrations in units of weight percent. Specific guidelines for determining accuracy and precision using these techniques are provided in the appropriate sections of this method. In general, however, the accuracy of any technique is determined through analysis of calibration materials which are characterized by multiple independent techniques in order to provide an unbiased value for the analyte (asbestos) in question. The precision of any technique is determined by multiple analyses of the sample. The analyst is the detector for stereomicroscopic and PLM techniques, as opposed to gravimetric and XRD techniques, and therefore must be calibrated as an integral part of the procedure.

As in the qualitative analysis, the laboratory should determine its accuracy and precision for quantitative asbestos analysis according to the type of material analyzed and the technique used for analysis. For example, the laboratory may determine that its analysts have a problem with calibrated area estimates of samples containing cellulose and chrysotile and therefore needs to make or find special calibration materials for this class of sample.

Calibration materials for quantitative analysis of asbestos are available through the Bulk Asbestos NVLAP as proficiency testing materials for those laboratories enrolled in NVLAP. In a report provided following a test round, the concentration of asbestos in each sample is given in weight percent with 95%/95% tolerance limits, along with a description of the major matrix components. Materials from other round robin and quality assurance programs for asbestos analysis may not have been analyzed by independent techniques; the concentrations may represent consensus PLM results that could be significantly biased. Therefore, values from these programs should <u>not</u> be used as calibration materials for quantitative analysis.

Calibration materials for quantitative analysis can also be synthesized by mixing asbestos and appropriate matrix materials, as described in Appendix C of this method. These

materials are usually simplifications of "real world" samples; therefore the accuracy and precision determined from analysis of these materials are probably ideal.

Limits on permissible analytical variability must be established by the laboratory prior to QC implementation. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed.) The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%. Quantitative results for standards including the mean and error estimate (typically 95% confidence or tolerance intervals) should be recorded. Over time these data can be used to help determine control limits for quality control charts.

The establishment and use of control charts is extensively discussed elsewhere in the literature. [1,2,3,4,5] Several cautions are in order:

- Control charts are based on the assumption that the data are distributed normally. Using rational subgrouping, the means of the subgroups are approximately normally distributed, irrespective of the distribution of the individual values in the subgroups. Control charts for asbestos analysis are probably going to be based on individual measurements, not rational subgroups. Check the data for normality before proceeding with the use of control charts. Ryan[8] suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.

- Include both prepared slides as well as bulk samples in your reference inventory.

- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

Data on analytical variability can be obtained by having analysts repeat their analyses of samples and also by having different analysts analyze the same samples.

3.3 Interlaboratory Quality Control

The establishment and maintenance of an interlaboratory QC program is fundamental to continued assurance that the data produced within the laboratory are of consistent high quality. Intralaboratory programs may not be as sensitive to accuracy and precision error, especially if the control charts (see Section 3.2.2) for all analysts in the laboratory indicate small percent differences. A routine interlaboratory testing program will assist in the detection of internal bias and analyses may be performed more frequently than proficiency

testing. Arrangements should be made with at least two (preferably more) other laboratories that conduct asbestos identification by PLM. Samples (the number of which is left to the participating laboratories, but at least 4-10) representing the types of samples and matrices routinely submitted to the lab for analysis should be exchanged with sufficient frequency to determine intralaboratory bias. Both reference slides and bulk samples should be used. Results of the interlaboratory testing program should be evaluated by each of the participating laboratories and corrective actions, if needed, identified and implemented. Since quantitation problems are more pronounced at low concentrations ($\leq$ 5%), it would be prudent to include approximately 30-50% from this concentration range in the sample selection process.

3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP[9] for bulk asbestos laboratories (PLM) does not use concensus quantitation results. Results are reported in weight percent with a 95% tolerance interval. The American Industrial Hygiene Association (AIHA)[10] also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

3.5 Systems Audits

Where performance audits are quantitative in nature, systems audits are qualitative. Systems audits are assessments of the laboratory quality system as specified in the Laboratory

Quality Assurance Manual. Such an audit might consist of an evaluation of some facet of the QA Manual, or the audit may be larger in scope. For example, the auditor might request specific laboratory data sheets which will be evaluated against written procedures for data recording in the laboratory. Or, the auditor might request air monitoring or contamination control data to review for frequency of sampling, analysis methodology, and/or corrective actions taken when problems were discovered. The audit report should reflect the nature of the audit as well as the audit results. Any recommendations for improvement should also be reflected in such a report.

3.6 References

1. **Quality Assurance for Air Pollution Measurement Systems. Volume I, Principles.** EPA-600/9-76-005, March, 1976.

2. Juran, J. and F. Gryna, **Quality Planning Analysis,** 2nd edition, McGraw-Hill, Inc., 1980.

3. Taylor, J.R., **Quality Control Systems,** McGraw Hill, Inc., 1989.

4. Ratliff, T.A., **The Laboratory Quality Assurance System,** Van Nostrand Reinhold, 1990.

5. Taylor, J.K., **Quality Assurance of Chemical Measurements,** Lewis Publishers, 1987.

6. Bulk Asbestos Handbook, National Institute of Standards and Technology, National Voluntary Laboratory Accreditation Program, NISTIR 88-3879, October 1988.

7. Harvey, B.W., "Classification and Identification Error Tendencies in Bulk Insulation Proficiency Testing Materials," **American Environmental Laboratory, 2**(2), 4/90, pp. 8-14.

8. Ryan, T.P., **Statistical Techniques for Quality Improvement,** John Wiley & Sons, Inc., New York, 1989.

9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.

10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.

APPENDIX A

Glossary Of Terms

APPENDIX A. GLOSSARY OF TERMS

Accuracy The degree of agreement of a measured value with the true or expected value.

Anisotropic Refers to substances that have more than one refractive index (e.g. are birefringent), such as nonisometric crystals, oriented polymers, or strained isotropic substances.

Asbestiform (morphology) Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5μm. Aspect ratios should be determined for fibers, not bundles.
- Very thin fibrils, usually less than 0.5 micrometers in width, and
- Two or more of the following:

  Parallel fibers occurring in bundles,

  Fiber bundles displaying splayed ends,

  Matted masses of individual fibers, and/or

  Fibers showing curvature

These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of $<$20:1 for fibers longer than 5μm. If a sample contains a fibrous component of which most of the fibers have aspect ratios of $<$20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

Asbestos - A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), and asbestiform anthophyllite, asbestiform tremolite, and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability, and 5) high heat resistance.

Becke Line - A band of light seen at the periphery of a specimen when the refractive indices of the specimen and the mounting medium are different; it is used to determine refractive index.

Bias - A systematic error characterized by a consistent (non-random) measurement error.

Binder - With reference to a bulk sample, a component added for cohesiveness (e.g. plaster, cement, glue, etc.).

Birefringence - The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated, using a Michel-Levy chart, from the interference colors observed under crossed polarizers. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below.

Qualitative	Quantitative(N-n)
none	0.00 or isotropic
low	≤ 0.010
moderate	0.011-0.050
high	> 0.050

Bulk Sample - A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and nonfriable materials.

Bundle - Asbestos structure consisting of several fibers having a common axis of elongation.

Calibration Materials - Materials, such as known weight % standards, that assist in the calibration of microscopists in terms of ability to quantitate the asbestos content of bulk materials.

Color - The color of a particle or fiber when observed in plane polarized light.

Compensator - A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530-550nm retardation).

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Detection Limit The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Dispersion Staining (focal masking) An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; ir it is used to determine refractive indices.

Error Difference between the true or expected value and the measured value of a quantity or parameter.

Extinction The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique, symmetrical and undulose.

Extinction Angle For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

Fiber With reference to asbestiform morphology, a structure consisting of one or more fibrils.

Fibril The individual unit structure of fibers.

Friable Refers to the cohesiveness of a bulk material, indicating that it may be crumbled or disaggregated by hand pressure.

Gravimetry Any technique in which the concentration of a component is determined by weighing. As used in this document, it refers to measurement of asbestos-containing residues after sample treatment by ashing, dissolution, etc.

Homogeneous Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

Heterogeneous Lacking uniformity in composition and/or distribution of material; components not uniform. Does not satisfy the conditions stated for homogenous; e.g., layered or in clumps, very coarse grained, etc.

Isotropic Refers to substances that have a single refractive index such as unstrained glass, un-oriented polymers and unstrained substances in the isometric crystal system.

Lamda Zero (λ_0) The wavelength (λ_0) of the dispersion staining color shown by a specimen in a medium; both the specimen and medium have the same refractive index at that wavelength.

Matrix Nonasbestos, nonbinder components of a bulk material. Includes such components as cellulose, fiberglass, mineral wool, mica, etc.

Michel-Levy Scale of Retardation colors A chart plotting the relationship between birefringence, retardation and thickness of anisotropic substances. Any one of the three variables can be determined if the other two are known.

Morphology The structure and shape of a particle. Characterization may be descriptive (platy, rod-like, acicular, etc) or in terms of dimensions such as length and diameter (see asbestiform).

Pleochroism The change in color or hue of colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

Point Counting A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to nonasbestos sample components.

Polarization Colors Interference colors displayed by anisotropic substances between two polarizers. Birefringence, thickness and orientation of the material affect the colors and their intensity.

Precision The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the variability of results.

Reference Materials Bulk materials, both asbestos-containing and nonasbestos-containing, for which the components are well-documented as to identification and quantitation.

Refractive Index (index of refraction) The ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as n and varies with wavelength and temperature.

Sign of Elongation Referring to the location of the high and low refractive indices in an elongated anisotropic substance, a specimen is described as positive when the higher refractive index is lengthwise (length slow), and as negative when the lower refractive index is lengthwise (length fast).

Standard Reference Material (SRM) A reference material certified and distributed by the National Institute of Standards and Technology.

Visual Estimate An estimation of concentration of asbestos in a sample as compared to the other sample components. This may be a volume estimate made during stereomicroscopic examination and/or a projected area estimation made during microscopic (PLM) examination.

APPENDIX B

Apparatus For Sample Preparation And Analysis

B1.0 INTRODUCTION

The following lists the apparatus and materials required and suggested for the methods of sample preparation and analysis described in the test method.[1,2,3]

B2.0 STEREOMICROSCOPIC EXAMINATION

The following are suggested for routine stereomicroscopic examination.

- HEPA-filtered hood or class 1 biohazard hood, negative pressure
- Microscope: binocular microscope, preferably stereoscopic, 5-60X magnification (approximate)
- Light source: incandescent or fluorescent
- Tweezers, dissecting needles, scalpels, probes, etc. (for sample manipulation)
- Glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. (sample containers)

The following are suggested for sample preparation.

- Mortar and pestle, silica or porcelain-glazed
- Analytical balance (readability less than or equal to one milligram) (optional)
- Mill or blender (optional)

B3.0 POLARIZED LIGHT MICROSCOPY

The laboratory should be equipped with a polarized light microscope (preferably capable of Köhler or Köhler-type illumination if possible) and accessories as described below.

- Ocular(s) binocular or monocular with cross hair reticle, or functional equivalent, and a magnification of at least 8X
- 10X, 20X, and 40X objectives, (or similar magnification)

- Light source (with optional blue "day-light" filter)
- 360-degree rotatable stage
- Substage condenser with iris diaphragm
- Polarizer and analyzer which can be placed at 90 degrees to one another, and can be calibrated relative to the cross-line reticle in the ocular.
- Accessory slot for wave plates and compensators (or demonstrated equivalent).
- Wave retardation plate (Red I compensator) with approximately 550 nanometer retardation, and with known slow and fast vibration directions.
- Dispersion staining objective or a demonstrated equivalent. (optional)
- Monochromatic filter (n_D), or functional equivalent. (optional)

In addition, the following equipment, materials and reagents are required or recommended.[1]

- NIST traceable standards for the major asbestos types (NIST SRM 1866 and 1867)
- Class I biohazard hood or better (see "Note", Section 2.2.5)
- Sampling utensils (razor knives, forceps, probe needles, etc.)
- Microscope slides and cover slips
- Mechanical Stage
- Point Counting Stage (optional)
- Refractive index liquids: 1.490-1.570, 1.590-1.720 in increments of less than or equal to 0.005; high dispersion, (HD) liquids are optional; however, if using dispersion staining, HD liquids are recommended.
- Mortar and pestle
- Distilled water
- HCl, ACS reagent grade concentrated

- Muffle furnace (optional)
- Mill or blender (optional)
- Beakers and assorted glassware (optional)
- Other reagents (tetrahydrofuran, amyl acetate, acetone, sodium hexametaphosphate, etc.) (optional)

B4.0 GRAVIMETRY

The following equipment, materials, and reagents are suggested.

- Scalpels
- Crucibles, silica or porcelain-glazed, with lids
- Muffle furnace temperature range at least to 500°C, temperature stable to $\pm$ 10°C, temperature at sample position calibrated to $\pm$ 10°C
- Filters, 0.4 μm pore size polycarbonate
- Petri dishes
- Glass filtration assembly, including vacuum flask, water aspirator, and/or air pump
- Analytical balance, readable to 0.001 gram
- Mortar and pestle, silica or porcelain-glazed
- Heat lamp or slide warmer
- Beakers and assorted glassware
- Centrifuge, bench-top
- Class I biohazard hood or better
- Bulb pipettes
- Distilled water
- HCl, reagent-grade concentrated

- Organic solvents (tetrahydrofuran, amyl acetate,etc)
- Ultrasonic bath

B5.0 X-RAY DIFFRACTION

Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- Mortar and pestle: agate or porcelain
- Razor blades
- Sample mill: SPEX, Inc., freezer mill or equivalent
- Bulk sample holders
- Silver membrane filters: 25-mm diameter, 0.45-μm pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006
- Microscope slides
- Vacuum filtration apparatus: Gelman No. 1107 or equivalent, the side-arm vacuum flask
- Microbalance
- Ultrasonic bath or probe: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent
- Volumetric flasks: 1-L volume
- Assorted pipets
- Pipet bulb
- Nonserrated forceps
- Polyethylene wash bottle
- Pyrex beakers: 50-mL volume

- Desiccator
- Filter storage cassettes
- Magnetic stirring plate and bars
- Porcelain crucibles
- Muffle furnace or low temperature asher
- Class 1 biohazard hood or better

Sample Analysis

Sample analysis requirements include an x-ray diffraction unit, equipped with:

- Constant potential generator; voltage and mA stabilizers
- Automated diffractometer with step-scanning mode
- Copper target x-ray tube: high intensity; fine focus, preferably
- X-ray pulse height selector
- X-ray detector (with high voltage power supply): scintillation or proportional counter
- Focusing graphite crystal monochromator; or nickel filter (if copper source is used, and iron fluorescence is not a serious problem)
- Data output accessories:
  Strip chart recorder
  Decade scaler/timer
  Digital printer

  or

  PC, appropriate software and Laser Jet Printer
- Sample spinner (optional)
- Instrument calibration reference specimen: α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

Reagents, etc.

Reference Materials The list of reference materials below is intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- Chrysotile: UICC Canadian, NIST SRM 1866 (UICC reference material available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK); (NIST Standard Reference Materials available from the National Institute of Standards and Technology, Office of Reference Standards, Gaithersburg, MD 20899)

- Crocidolite: UICC, NIST SRM 1866.

- "Amosite": UICC, NIST SRM 1866.

- Anthophyllite-Asbestos: UICC, NIST SRM 1867

- Tremolite Asbestos: Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 900064 (washed with dilute HCl to remove small amount of calcite impurity); Indian tremolite, Rajasthan State, India; NIST SRM 1867.

- Actinolite Asbestos: NIST SRM 1867

Adhesive Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

Surfactant 1 Percent aerosol OT aqueous solution or equivalent.

Isopropanol ACS Reagent Grade.

B6.0 ANALYTICAL ELECTRON MICROSCOPY

AEM equipment requirements will not be discussed in this document; it is suggested that equipment requirements stated in the AHERA regulations be followed. Additional information may be found in the NVLAP Program Handbook for Airborne Asbestos Analysis.[3]

The following additional materials and equipment are suggested:

- Analytical balance, readable to 0.001 gram
- Ultrasonic bath
- Glass filtration assembly (25mm), including vacuum flask and water aspirator
- Mixed cellulose ester (MCE) filters (0.22μm pore size) or 0.2μm pore size polycarbonate filters
- MCE backing filters (5μm pore size)
- Silica mortar and pestle
- Beakers glass and disposable
- Pipettes, disposable, 1,5, and 10 ml

B7.0 REFERENCES

1. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Bulk Asbestos Handbook, NISTIR 88-3879, 1988.
2. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples**, U.S. E.P.A. 600/M4-82-020, 1982.
3. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Program Handbook for Airborne Asbestos Analysis, NISTIR 89-4137, 1989.

APPENDIX C

Preparation and Use of Bulk Asbestos
Calibration Standards

C1.0 INTRODUCTION

Evaluation of the results from national proficiency testing programs for laboratories analyzing for asbestos in bulk materials indicates that laboratories have had, and continue to have, problems with quantitation of asbestos content, especially with samples having a low asbestos concentration.[1] For such samples, the mean value of asbestos content reported by laboratories may be four to ten times the true weight percent value. It is assumed that the majority of the laboratories quantify asbestos content by visual estimation, either stereomicroscopically or microscopically; therefore, the problem of quantitation must be attributed to lack of or inadequate calibration of microscopists.

As calibration standards for asbestos-containing bulk materials are not currently commercially available, laboratories should consider generating their own calibration materials. This may be done rather easily and inexpensively.

C2.0 MATERIALS AND APPARATUS

Relatively pure samples of asbestos minerals should be obtained. Chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867) are available from NIST. A variety of matrix materials are commercially available; included are calcium carbonate, perlite, vermiculite, mineral wool/fiberglass, and cellulose. Equipment, and materials needed to prepare calibration bulk materials are listed below.

- Analytical balance, readable to 0.001 gram
- Blender/mixer; multi-speed, ~ one quart capacity
- Filtration assembly, including vacuum flask, water aspirator and/or air pump (optional)
- HEPA-filtered hood with negative pressure
- Filters, 0.4μm pore size polycarbonate (optional)
- Beakers and assorted glassware, weigh boats, petri dishes, etc.
- Hot/warm plate

- Asbestos minerals
- Matrix materials
- Distilled water.

C3.0 MATERIAL FORMULATION PROCEDURES

The formulation procedure involves first weighing appropriate quantities of asbestos and matrix material to give the desired asbestos weight percent. The following formula may be used to determine the weights of asbestos and matrix materials needed to give a desired weight percent asbestos.

$$\frac{WTa}{Wa} = \frac{WTm}{Wm}$$

Where:

WTa = weight of asbestos in grams (to 0.001 gram)
WTm = weight of matrix materials in grams (to 0.001 gram)
Wa = weight percent asbestos
Wm = weight percent matrix

Example: The desired total weight for the calibration sample is ~ 10 grams containing 5% asbestos by weight. If 0.532 grams of asbestos are first weighed out, what corresponding weight of matrix material is required?

WTa = 0.532 grams
Wa = 5%
Wm = 95%

$$\frac{0.532}{5} = \frac{WTm}{95}$$

Then: WTm = 10.108 grams

The matrix is then placed into the pitcher of a standard over-the-counter blender, the pitcher being previously filled to approximately one-fourth capacity (8-10 ounces) with distilled water. Blending is performed at the lowest speed setting for approximately ten seconds which serves to disaggregate the matrix material. The asbestos is then added, with additional blending of approximately 30 seconds, again at the lowest speed setting. Caution should be taken not to overblend the asbestos-matrix mixture. This could result in a significant reduction in the size of the asbestos fibers causing a problem with detection at normal magnification during stereomicroscopic and microscopic analyses. Ingredients of the

pitcher are then poured into a filtering apparatus, with thorough rinsing of the pitcher to ensure complete material removal. After filtering, the material is transferred to a foil dish which is placed on a hot plate. The material is covered and allowed to sit over low heat until drying is complete; intermittent stirring will speed the drying process. For fine-grained matrix materials such as gypsum, calcium carbonate, clays, etc., the sample is not filtered after the blending process. Instead, the ingredients in the pitcher are transferred into a series of shallow, glass (petri) dishes. The ingredients should be stirred well between each pouring to minimize the possible settling (and over-representation) of some components. The dishes are covered and placed on a hot plate until the contents are thoroughly dried. For small quantities of any matrix materials (15 grams or less), air-drying without prior filtering is generally very suitable for removing water from the prepared sample. For each material, the final step involves placing all formulated, dried subsamples into a plastic bag (or into one petri dish, for small quantities), where brief hand-mixing will provide additional blending and help to break up any clumps produced during drying. **All operations should be performed in a safety-hood with negative pressure.**

C4.0 ANALYSIS OF MATERIALS

All formulations should be examined with the stereomicroscope to determine homogeneity. Gravimetric analysis (ashing and/or acid dissolution) should be performed on those materials containing organic and/or acid-soluble components. Matrix materials to which no asbestos has been added should be analyzed by gravimetric analysis to determine the amount of nonashable or insoluble materials that are present. Several subsamples of each material should be analyzed by the gravimetric technique to provide information concerning the uniformity of the prepared materials. Experience has shown that the previously described formulation procedure results in relatively homogeneous materials.[2]

C4.1 Stereomicroscopic Analysis

Visual estimation of sample components using the stereomicroscope is in reality a comparison of the relative volumes of the components.[3] Therefore, differences in specific gravity between asbestos and matrix material must be considered and the relationship

between weight percent and volume percent must be determined.[4] Materials such as expanded vermiculite, perlite, and cellulose have specific gravities significantly lower than asbestos minerals. Table C1 lists the specific gravities for the three most commonly encountered asbestos varieties and several common matrix materials.

TABLE C1. SPECIFIC GRAVITIES OF ASBESTOS VARIETIES AND MATRIX MATERIALS

Asbestos Type	Specific Gravity	Matrix Type	Specific Gravity
Chrysotile	2.6	Calcium Carbonate	2.7
		Gypsum	2.3
Amosite	3.2	Perlite	~0.4
		Vermiculite (expanded)	~0.3
		Mineral Wool	~2.5
Crocidolite	3.3	Fiberglass	~2.5
		Cellulose	~0.9

The conversion of weight percent asbestos to equivalent volume percent asbestos is given by the following formula:

$$\frac{\frac{Wa}{Ga}}{\frac{Wa}{Ga} + \frac{Wm}{Gm}} \times 100 = Va$$

where:

Wa = weight percent asbestos
Ga = specific gravity of asbestos
Wm = weight percent matrix
Gm = specific gravity of matrix
Va = volume percent asbestos

Example: Chrysotile and perlite have been combined to form a 5% asbestos calibration standard, by weight. What is the equivalent volume percent asbestos?

$$Wa = 5\% \quad Ga = 2.6 \quad Wm = 95\% \quad Gm = 0.4$$

$$Va = \frac{\frac{5}{2.6}}{\frac{5}{2.6} + \frac{95}{0.4}} \times 100 = 0.8\%$$

Conversely, to convert volume percent asbestos to equivalent weight percent, the following formula may be used.

$$\frac{(Va)(Ga)}{(Va)(Ga)+(Vm)(Gm)} \times 100 = Wa$$

Vm = volume percent matrix

Example: A calibration standard consisting of amosite and cellulose is estimated to contain 2% asbestos, by volume. What is the equivalent weight percent asbestos?

$$Va = 2\% \quad Ga = 3.2 \quad Vm = 98\% \quad Gm = 0.9$$

$$Wa = \frac{(2)(3.2)}{(2)(3.2)+(98)(0.9)} \times 100 = 6.77\%$$

Volume percentages should be calculated for all calibration materials prepared so that visual estimates determined by examination with the stereomicroscope may be compared to true volume concentrations.

Figure C1 illustrates the relationship between volume percent and weight percent of chrysotile mixed with vermiculite and cellulose respectively. It should be noted that when asbestos in a low weight percentage is mixed with matrix materials having low specific gravities (vermiculite, perlite), the resulting volume concentration of asbestos is very low For example, a mixture containing three percent chrysotile by weight in a cellulose matrix would result in a volume percent asbestos of approximately 1.1%; in a vermiculite matrix, the resulting volume percent asbestos would be approximately 0.4%. In the latter case especially, an analyst might possibly fail to detect the asbestos or consider it to be present in only trace amounts.

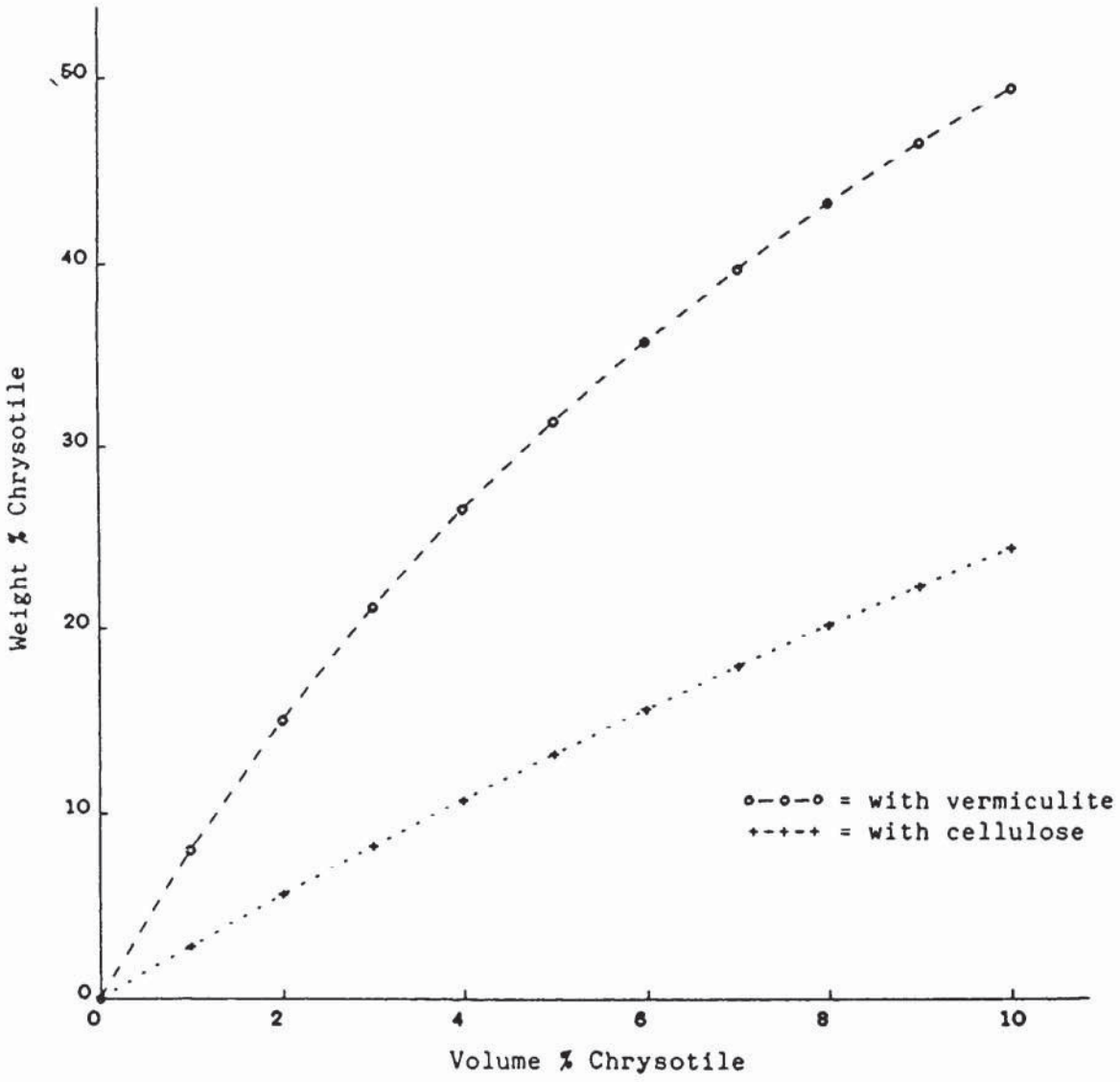


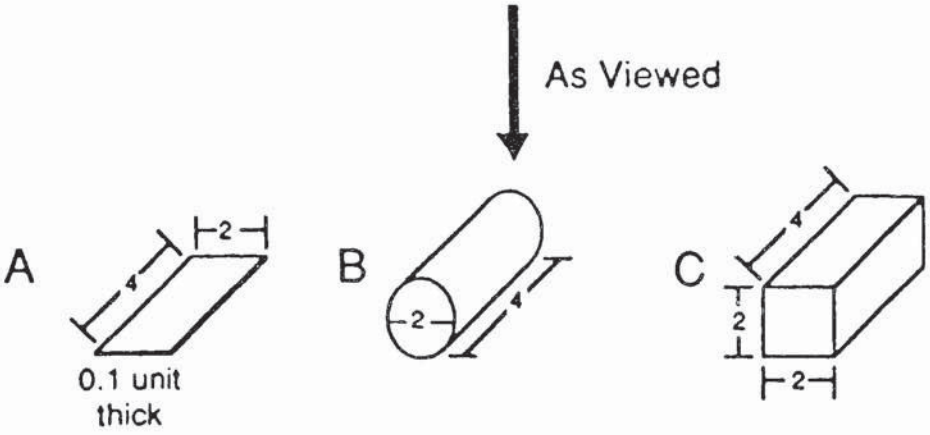
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

C4.2 Microscopical Analysis (PLM)

The polarized light microscope may be used to quantify asbestos and other components of a sample. Slide mounts are prepared from "pinch" samples of the calibration material and asbestos content is determined by visual area estimate and/or point counting. Both of these quantitation techniques are in fact estimates or measurements of the relative projected areas of particles as viewed in two dimensions on a microscope slide. For quantitation results to be meaningful, the following conditions should be met:

- The sample should be homogeneous for slide preparations, which are made from small pinches of the sample, to be representative of the total sample.
- Slide preparation should have an even distribution of particles and approach a one particle thickness (seldom achieved) to avoid particle overlap.
- All materials used should be identified and specific gravities determined in order to relate area percent to volume and/or weight percent.
- The size (thickness) relationship between matrix particles and asbestos fibers should be determined if the results based on projected area are to be related to volume and/or weight percent.

Particle characteristics can greatly affect the quantitation results obtained by visual area estimation or point counting. Figure C2 illustrates three hypothetical particle shapes of identical length and width (as viewed from above). Although the three-dimensional shape is different, the projected area is equal for all particles. The table accompanying Figure C2 presents data for each particle in terms of thickness, volume and projected area. It should be noted that although the projected areas may be equal, the volumes represented by the particles may vary by a factor of 20(0.8 vs 16 cubic units). It is obvious that quantitation of a sample consisting of a mixture of particles with widely ranging particle thicknesses could result in different results. For example, if a sample contained relatively thick bundles of asbestos and a fine-grained matrix such as clay or calcium carbonate, the true asbestos content (by volume) would likely be underestimated. Conversely, if a sample contained thick "books" of mica and thin bundles of asbestos, the asbestos content (by volume) would likely be overestimated.



Particle	Thickness	Volume	Projected Area
A	0.1 units	0.8 cubic units	8 sq. units
B	2 units	12.6 cubic units	8 sq. units
C	2 units	16 cubic units	8 sq. units

Note that although all particles have the same projected area, particle C volume is 20x that of particle A.

Figure C2. Relationship of projected area to volume and thickness for three different particles as viewed on a slide mount.

Table C2 illustrates several examples of expected results from area estimates or point counting of samples in which the asbestos fibers and matrix particles differ in thickness.

TABLE C2. RELATIONSHIP OF WEIGHT PERCENT, VOLUME PERCENT AND PARTICLE THICKNESS TO QUANTITATION RESULTS

Composition of Sample In Wt. %	Theoretical Vol. % Asbestos	Thickness Factor* (Matrix/Asbestos)	Expected Area %
1% Amosite 99% Calcium Carbonate	0.9	0.5	0.4
1% Amosite 99% Calcium Carbonate	0.9	1	0.9
1% Amosite 99% Calcium Carbonate	0.9	2	1.8
1% Amosite 99% Vermiculite	0.1	1	0.1
1% Amosite 99% Vermiculite	0.1	10	1.0
1% Amosite 99% Vermiculite	0.1	20	2.0
1% Amosite 99% Vermiculite	0.1	30	2.9

* Value represents the relationship between the mean thickness of the matrix particles compared to the mean thickness of the asbestos particles.

It should be noted that it is not uncommon for matrix particle thickness to differ greatly from asbestos fiber thickness, especially with matrix materials such as vermiculite and perlite; vermiculite and perlite particles may be 20 - 30 times as thick as the asbestos fibers.

The general size relationships between matrix particles and asbestos fibers may be determined by scanning slide mounts of a sample. A micrometer ocular enables the microscopist to actually measure particle sizes.

If a thickness factor can be determined for a calibration sample of known volume proportions of asbestos and matrix materials, an expected equivalent projected area asbestos can be calculated using the following formula:

$$\frac{Va}{\frac{Vm}{T} + Va} \times 100 = Aa$$

where:

Va = true volume percent asbestos
Vm = true volume percent matrix
T = thickness factor (mean size matrix particle/mean size asbestos fiber)
Aa = expected projected area percent asbestos

Example: A calibration standard of known weight percent asbestos is determined, by factoring in component specific gravities, to be 5.0% asbestos by volume. The matrix particles are estimated to be ten times thicker than the asbestos fibers. What would be the expected projected area percentage of asbestos?

Va = 5%
Vm = 95%
T = 10

$$Aa = \frac{5}{\frac{95}{10} + 5} \times 100 = 34.5\%$$

Conversely, to convert projected area percent asbestos to equivalent volume percent, the following formula may be used:

$$\frac{Aa}{T(Am) + Aa} \times 100 = Va$$

Where: Am = projected area matrix

Example: A slide containing a subsample of an amosite/mineral wool calibration standard is determined by point counting to have a projected area asbestos of 18.6%. If the mineral wool fibers are estimated to be six times the asbestos fibers, in diameter, what is the equivalent volume percent asbestos?

$$Am = 81.4\%$$
$$Aa = 18.6\%$$
$$T = 6$$

$$Va = \frac{(18.6)}{6(81.4) + 18.6} \times 100 = 3.67\%$$

Based on specific gravity values listed in Table 1C and on the above volume asbestos determination, what is the equivalent weight percent asbestos in the sample?

$$Va = 3.67\%$$
$$Ga = 3.2$$
$$Vm = 96.33\%$$
$$Gm = 2.5$$

$$Wa = \frac{(3.67)(3.2)}{(3.67)(3.2) + (96.33)(2.5)} \times 100 = 4.7\%$$

C5.0 USE OF CALIBRATION STANDARDS FOR QA/QC

Once the materials have been formulated and thoroughly characterized by all techniques to determine their suitability as calibration standards, a system for incorporating them into the QA/QC program should be established. Someone should be designated (QA officer, lab supervisor, etc.) to control the distribution of standards and to monitor the analysis results of the microscopists. Both precision and accuracy may be monitored with the use of suitable standard sets.

Records such as range charts, control charts, etc. may be maintained for volume (stereomicroscopic estimates), area (PLM) estimates and point counts. For point counts and area estimates, relatively permanent slides may be made using epoxy or Melt Mount *. Such slides may be very accurately quantified over time as to point count values, and due to their very long shelf life, may be used for QA/QC purposes almost indefinitely.

C6.0 REFERENCES

1. "Analysis Summaries for Samples used in NIST Proficiency Testing", National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) for Bulk Asbestos, January 1989 to present.

2. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991.

3. Perkins, R. L. and M. E. Beard, "Estimating Asbestos Content of Bulk Materials", **National Asbestos Council Journal**, Vol. 9, No. 1, 1991, pp. 27-31.

4. **Asbestos Content in Bulk Insulation Samples: Visual Estimates and Weight Composition**, U.S. Environmental Protection Agency 560/5-88-011, 1988.

APPENDIX D

Special-Case Building Materials

Asbestos laboratories are now called upon to analyze many types of bulk building materials that are very difficult to characterize by routine PLM analysis. These materials are dominantly nonfriable and can be grouped into the following categories:

- Cementitious Products (pipe, sheeting, etc.)
- Viscous Matrix Products (adhesives, cements, coatings, etc.)
- Vinyl Materials (vinyl floor tile, sheeting)
- Asphaltic Roofing Materials (shingles, roll roofing)
- Miscellaneous Products (paints, coatings, friction plates, gaskets, etc.)

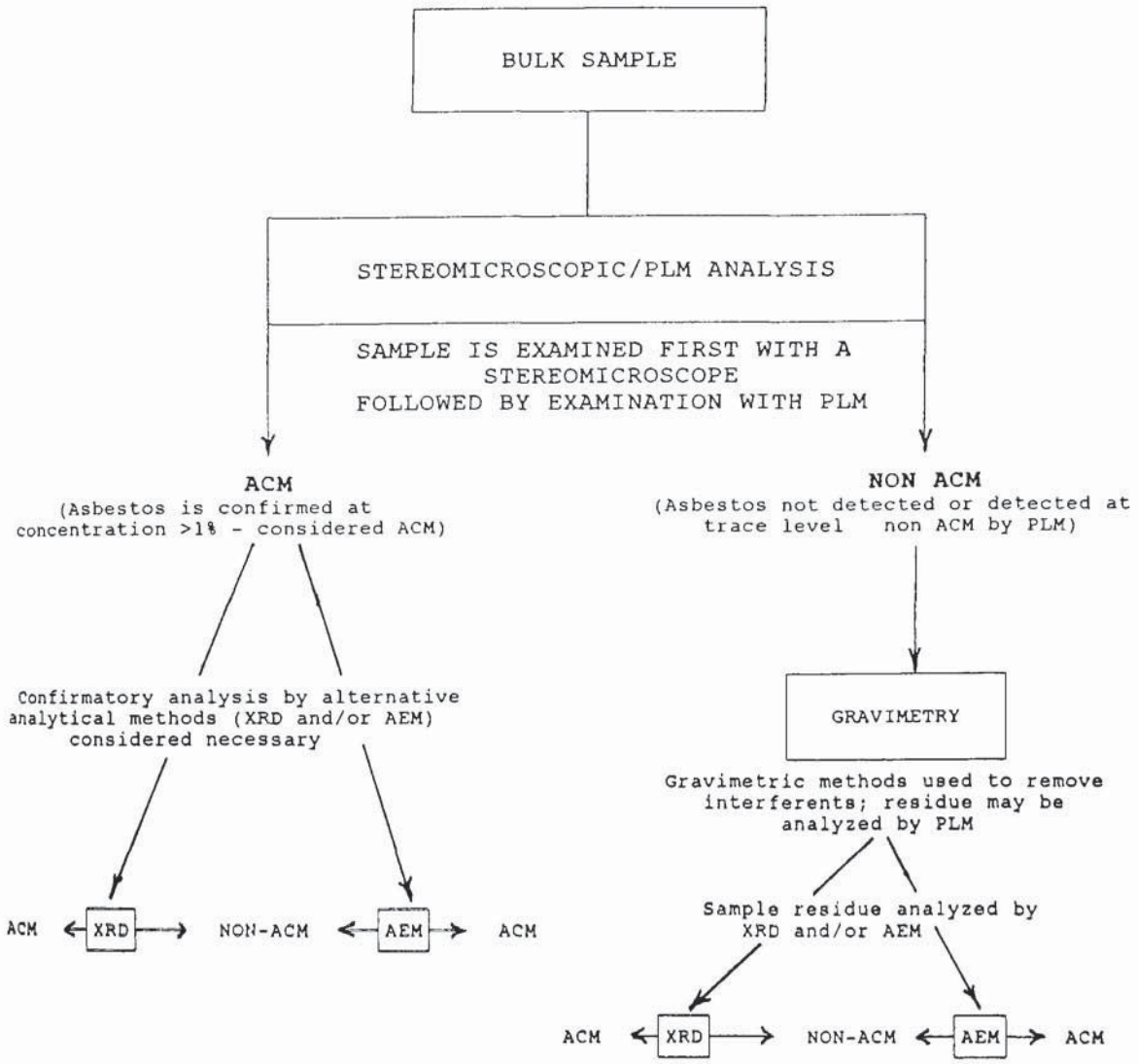
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components (see Section 2.3, Gravimetry). Materials containing an appreciable amount of calcareous material may be treated by dissolution with hydrochloric acid. Samples containing organic binders such as vinyl, plasticizers, esters, asphalts, etc. can be treated with organic solvents or ashed in a muffle furnace (preferred method) or low temperature plasma asher to remove unwanted components. Materials containing cellulose, synthetic organic fibers, textiles, etc. may also be ashed in a muffle furnace or low temperature plasma asher.

The method chosen for analysis of a sample after treatment is dependent on asbestos concentration and/or fiber size. An examination of the sample residue by PLM may disclose asbestos if the fibers are large enough to be resolved by the microscope, but additional analytical methods are required if the sample appears negative. Analysis by XRD is not fiber-size dependent, but may be limited by low concentration of asbestos and the presence of interfering mineral phases. In addition, the XRD method does not differentiate between fibrous and nonfibrous varieties of a mineral. Analysis by AEM is capable of providing positive identification of asbestos type(s) and semi-quantitation of asbestos content.

The following flowchart illustrates a possible scheme for the analysis of special-case building materials.

NOTE: Preliminary studies indicate that the XRD method is capable of detecting serpentine (chrysotile) in floor tile samples without extensive sample preparation prior to XRD analysis. XRD analysis of small, intact sections of floor tile yielded diffraction patterns that confirmed the presence of serpentine, even at concentrations of ~one percent by weight. TEM analysis of these same tiles confirmed the presence of chrysotile asbestos. With further investigation, this method may prove applicable to other types of nonfriable materials.

FLOWCHART FOR QUALITATIVE ANALYSIS OF SPECIAL CASE BUILDING MATERIALS SUCH AS FLOOR TILES, ASPHALTIC MATERIALS, VISCOUS MATRIX MATERIALS, ETC.*



*Although this flowchart is applicable to all bulk materials, it is primarily intended to be used with known problem materials that are difficult to analyze by PLM due to low asbestos concentration, and/or small fiber size, and/or interfering binder/matrix. In addition to being qualitative, the results may also be semi-quantitative. It should not be assumed that all samples need to be analyzed by AEM and XRD. The flowchart simply illustrates options for methods of analysis. Alternate methods such as SEM may be applicable to some bulk materials.

*U S GOVERNMENT PRINTING OFFICE 1993 -750 -002/ 80237

Designation: D 6480 – 05

Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Surface Loading by Transmission Electron Microscopy[1]

This standard is issued under the fixed designation D 6480; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to identify asbestos in samples wiped from surfaces and to provide an estimate of the concentration of asbestos reported as the number of asbestos structures per unit area of sampled surface. The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fiber aggregates.

1.2 This test method describes the equipment and procedures necessary for wipe sampling of surfaces for levels of asbestos structures. The sample is collected onto a particle-free wipe material (wipe) from the surface of a sampling area that may contain asbestos.

1.2.1 The collection efficiency of this wipe sampling technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, and other factors.

1.2.2 This test method is generally applicable for an estimate of the surface loading of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.3 Asbestos identification by transmission electron microscopy (TEM) is based on morphology, electron diffraction (ED), and energy dispersive X-ray analysis (EDXA).

1.4 This test method allows determination of the type(s) of asbestos fibers present.

1.4.1 This test method cannot always discriminate between individual fibers of the asbestos and nonasbestos analogues of the same amphibole mineral.

1.4.2 There is no lower limit to the dimensions of asbestos fibers that can be detected. However, in practice, the lower limit to the dimensions of asbestos fibers, that can be detected, is variable and dependent on individual microscopists. Therefore, a minimum length of 0.5 μm has been defined as the shortest fiber to be incorporated in the reported results.

1.5 *This test method does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1193 Specification for Reagent Water

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D22

2.2 *Government Standard:*[3]

40 CFR 763, USEPA, Asbestos-Containing Materials in Schools: Final Rule and Notice, Appendix A to Sub-part E

2.3 *U.S. Environmental Protection Agency Standards:*[3]

EPA 600/4-83-043 Analytical Method for the Determination of Asbestos in Water

EPA 747-R-95-001 USEPA, Residential Sampling for Lead: Protocols for Dust and Soil Sampling: Final Report

3. Terminology

3.1 *Definitions*—For definitions of general terms used in this test method, refer to Terminology D 1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *amphibole asbestos*—amphibole in an asbestiform habit (**1**).[4]

3.2.2 *analytical sensitivity*—the calculated asbestos structure concentration in asbestos structures/square centimetre,

[1] This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

Current edition approved March 1, 2005. Published March 2005. Originally approved in 1999. Last previous edition approved in 1999 as D 6480 - 99.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

equivalent to counting of one asbestos structure in the analysis calculated using Eq 2.

3.2.3 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate minerals, that are easily separated into long, thin, flexible, strong fibers when crushed or processed **(1-3)**.

3.2.3.1 *Discussion*—Included in the definition are the asbestiform varieties of serpentine (chrysotile), riebeckite (crocidolite), grunerite (grunerite asbestos [Amosite]), anthophyllite (anthophyllite asbestos), tremolite (tremolite asbestos), and actinolite (actinolite asbestos). The amphibole mineral compositions are defined in accordance with nomenclature of the International Mineralogical Association **(3,4)**.

Asbestos	Chemical Abstracts Service Registry No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos [Amosite]	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.2.4 *asbestos structure*—a term applied to isolated fibers or to any connected or overlapping grouping of asbestos fibers or bundles, with or without other nonasbestos particles.

3.2.5 *aspect ratio*—the length to width ratio of a particle.

3.2.6 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.7 *camera length*—the equivalent projection length between the specimen and its selection diffraction pattern, in the absence of lens action.

3.2.8 *chrysotile*—a group of fibrous minerals of the serpentine group that have the nominal composition $Mg_3Si_2O_5(OH)_4$ and have the crystal structure of either clinochrysotile, orthochrysotile, or parachrysotile. Most natural chrysotile deviates little from this nominal composition. Chrysotile may be partially dehydrated or magnesium-leached both in nature and in building materials. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Chrysotile is the most prevalent type of asbestos.

3.2.9 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.10 *d-spacing or inter-planar spacing*—the perpendicular distance between identical adjacent and parallel planes of atoms in a crystal.

3.2.11 *electron diffraction*—techniques in electron microscopy that include selected area electron diffraction (SAED) and microdiffraction by which the crystal structure of a specimen is examined.

3.2.12 *energy dispersive X-ray analysis*—measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyzer system.

3.2.13 *eucentric*—the condition when the area of interest of an object is placed on a tilting axis at the intersection of the electron beam at that axis and is in the plane of focus.

3.2.14 *fiber*—an elongate particle with parallel or stepped sides. For the purposes of this test method, a fiber is defined to have an aspect ratio equal to or greater than 5:1 and a minimum length of 0.5 μm (see 40 CFR 763).

3.2.15 *fibril*—a single fiber, that cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.2.16 *fibrous mineral*—a mineral composed of parallel, radiating, or interlaced aggregates of fibers from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.17 *fibrous structure*—a fiber, or connected grouping of fibers, with or without other particles.

3.2.18 *field wipe blank*—a clean, unused, moistened wipe from the same supply that is used for sampling. Field wipes shall be processed in the same manner used to collect field samples with the exception that no surface is wiped. Each wipe designated as a field wipe should be removed from the bulk pack, moistened, and folded in the same manner as the field samples and placed in a sample container labeled as field wipe.

3.2.19 *filter blank*—an unused, unprocessed filter of the type used for liquid filtration.

3.2.20 *filtration blank*—a filter prepared from 250 mL of water.

3.2.21 *habit*—the characteristic crystal growth form or combination of these forms of a mineral, including characteristic irregularities.

3.2.22 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration. The particles are removed from the original medium and deposited on a second filter prior to analysis.

3.2.23 *limit of detection*—the limit of detection for a measurement by this test method is 2.99 multiplied by the analytical sensitivity for the measurement.

3.2.23.1 *Discussion*—This limit of detection is based on the assumption that the count resulting from potential filter contamination, sample preparation contamination, and other uncontrollable background sources is no greater than 0.05 structures per sample. At this time, however, this subcommittee has no empirical data to confirm this rate.

3.2.24 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by, a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition.

3.2.25 *process blank*—an unused wipe (that has not been taken into the field) processed in accordance with the entire preparation and analytical procedure.

3.2.26 *replicate sampling*—one of several identical procedures or samples.

[5] The nonasbestiform variations of the minerals indicated in 3.2.3.1 have different Chemical Abstract Service (CAS) numbers.

3.2.27 *serpentine*—a group of common rock-forming minerals having the nominal formula: $Mg_3Si_2O_5(OH)_4$. For further information see Ref. (**4**).

3.2.28 *structure*—a single fiber, fiber bundle, cluster, or matrix.

3.2.29 *structure number concentration*—concentration expressed in terms of asbestos structure number per unit of surface area.

3.2.30 *zone-axis*—the crystallographic direction of a crystal that is parallel to the intersecting edges of the crystal faces defining the crystal zone.

3.3 *Symbols*:

eV = electron volt
h = hour
J = joule
kV = kilovolt
min = minute(s)
mL = millilitre (10^{-3} litre)
μL = microlitre (10^{-6} litre)
mm = millimetre (10^{-3} metre)
μm = micrometre (10^{-6} metre)
nm = nanometre (10^{-9} metre)
s = second(s)
W = watt
Pa = pascals

3.4 *Acronyms*:

DMF = dimethyl formamide
ED = electron diffraction
EDXA = energy dispersive X-ray analysis
FWHM = full width, half maximum
HEPA = High Efficiency Particulate Air
MCE = mixed cellulose ester and also refers to pure cellulose nitrate filters
PC = polycarbonate
TEM = transmission electron microscope

4. Summary of Test Method

4.1 Wiping a surface of known area with a wipe material collects a sample. The sample is transferred from the wipe material to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane filter. A section of the membrane filter is prepared and transferred to a TEM grid, using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using ED and EDXA at a magnification from 15 000 to 20 000 ×.

5. Significance and Use

5.1 This wipe sampling and indirect analysis test method is used for the general testing of surfaces for asbestos. It is used to assist in the evaluation of surfaces in buildings, such as ceiling tiles, shelving, electrical components, duct work, and so forth. This test method provides an index of the concentration of asbestos structures per unit area sampled as derived from a quantitative measure of the number of asbestos structures detected during analysis.

5.1.1 This test method does not describe procedures or techniques required for the evaluation of the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos sampled from a surface and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information (for example, air sampling data) in their evaluation.

5.2 One or more large asbestos-containing particles dispersed during sample preparation may result in large asbestos surface loading results in the TEM analyses of that sample. It is, therefore, recommended that multiple replicate independent samples be secured in the same area, and that a minimum of three such samples be analyzed by the entire procedure.

6. Interferences

6.1 The following materials have properties (that is, chemical composition or crystalline structure) that are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials shall be maintained in the laboratory for comparison with asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 Antigorite,
6.1.2 Fibrous talc,
6.1.3 Halloysite,
6.1.4 Hornblende and other amphiboles,
6.1.5 Palygorskite (attapulgite),
6.1.6 Pyroxenes,
6.1.7 Sepiolite, and
6.1.8 Vermiculite scrolls.

7. Apparatus

7.1 *Equipment and Materials for Sampling*:

7.1.1 *Disposable Wet Towels.*

7.1.2 *Masking Tape.*

7.1.3 *Measuring Tape.*

7.1.4 *Powderless, Rubber Gloves.*

7.1.5 *Sample Container*, clean, sealable, used for transporting the sample to the laboratory.

7.1.6 *Template to Delineate Sampling Area*, a reusable or disposable template of nonparticle-shedding material, such as aluminum, plastic, or nonshedding cardboard. A variety of shapes (for example, square, rectangular) are acceptable. All templates shall have accurately known inside dimensions. Templates should be thin (less than ⅛ in. (3 mm)) and capable of lying flat on a flat surface. Clean reusable template before and after each use with a suitable cleaning method, such as surfactant solution or particle-free disposable wipe.

7.1.7 *Wipe*, particle free, sealed edge, continuous filament cloth sampling medium. Satisfactory brands are available through commercial scientific suppliers. This material is commonly listed under *clean room wiper*. Wipe brands or sources should not contain unacceptable particle or fiber levels. Prior to use, TEM analysis on blank wipe preparations should be performed to determine that background particle and fiber levels will not interfere with preparation and analysis.

7.2 *Equipment and Materials for Preparation*:

7.2.1 *Carbon Rod Electrodes*, spectrochemically pure for use in the vacuum evaporator during carbon coating of filters.

7.2.2 *Carbon Rod Sharpener*—An instrument used to sharpen carbon rod electrodes.

7.2.3 *Cork Borer*, 7-mm diameter.

7.2.4 *Disposable Tip Micropipettes*, 30 µL.

7.2.5 *Electron Microscope Grids (for example, Cu Au, Ni)*, 200 mesh TEM grids with grid openings of uniform size. Use grids with numerical or alphabetical indexing, or both, of individual grid openings to facilitate the relocation of individual grid openings for quality assurance purposes.

7.2.6 *Filtration Unit*, 25 or 47-mm filter funnel (either glass or disposable). Filter funnel assemblies, either glass or disposable plastic, using a 25 or 47-mm diameter filter.

7.2.7 *Graduated, Disposable Pipettes*, 1, 5, or 10–mL sizes, glass or plastic.

7.2.8 *Grid Box*, for electron microscope grid storage.

7.2.9 *High Efficiency Particulate Air (HEPA) Filtered Negative Flow Hood.*

7.2.10 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47-mm diameter, ≤ 0.22 and 5-µm pore size.

7.2.11 *pH Paper.*

7.2.12 *Plasma Asher*, for preparation of TEM specimens from MCE filters. The plasma asher shall have a radio frequency power rating of 50 W or higher and be provided with a controlled, filtered oxygen flow. Admission of filtered air shall be through a valve to control the speed of air admission so that rapid air admission does not disturb particulate matter from the surface of the filter after the etching step.

7.2.13 *Plastic Petri Dishes*, or similar container to retain filters (50 mm in diameter or larger). These petri dishes may be used as storage containers for archiving filters.

7.2.14 *Polycarbonate (PC) Membrane Filters*, 25 or 47-mm diameter ≤ 0.2-µm pore size.

7.2.15 *Routine Electron Microscopy Tools and Supplies*, such as fine-point tweezers or forceps, scalpel holders and blades, microscope slides, double-coated adhesive tape, gummed paper reinforcement rings, lens tissue, gold wire, tungsten filaments, and other routine supplies.

7.2.16 *Side Arm Filter Flask*, 1000 mL.

7.2.17 *Slide Warmer or Low Temperature Drying Oven*, for drying filters or heating slides during the preparation of TEM specimens from MCE or cellulose nitrate filters, capable of maintaining a temperature from 65 to 70°C.

7.2.18 *Specimen Bottle*, wide mouth, sealable, capable of accommodating the wipe and a minimum of approximately 500 mL of distilled water.

7.2.19 *Sputter Coater*, for deposition of gold onto TEM specimens to be used as an internal calibration of ED patterns. Other calibration materials are also acceptable. Experience has shown that a sputter coater allows control of the deposition thickness of the calibration material.

7.2.20 *Solvent Washer (Jaffe washer) (see EPA 600/4-83-043)*, allows for dissolution of the filter polymer while leaving an intact evaporated carbon film supporting the fibers and other particles from the filter surface. One design of a washer, that has been found satisfactory for various solvents and filter media, is shown in Fig. 1. Use dimethyl formamide or acetone for dissolving MCE or cellulose nitrate filters. Use either chloroform or 1-methyl-2-pyrrolidone, or a mixture of 20 % 1-2-diaminoethane and 80 % 1-methyl-2-pyrrolidone, or dissolving PC filters. The higher evaporation rates of chloroform and acetone require that a reservoir of 10 to 50 mL of solvent be used, that may need replenishment during the procedure. DMF and 1-methyl-2-pyrrolidone have lower vapor pressures, and much smaller volumes of solvent may be used. Use the washer in a fume hood, and keep the petri dishes covered with their lids when specimens are not being inserted or removed during the solvent dissolution. Clean the washer before it is used for each batch of specimens.

7.2.21 *Ultrasonic Bath*, table top model (100 W).

7.2.22 *Vacuum Coating Unit*, capable of producing a vacuum better than 0.013 Pa, used for vacuum deposition of carbon on the membrane filters. A sample holder that will allow a glass microscope slide to be tilted and continuously rotated during the coating procedure is recommended. A liquid nitrogen trap may be used to minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum coating unit may also be used for deposition of the thin film of gold, or other calibration material, when it is required on TEM specimens as an internal calibration of ED patterns.

7.2.23 *Vacuum Pump*, able to maintain a vacuum of at least 20 kPa.

7.3 *Equipment and Materials for Analysis*:

7.3.1 *Calibration Specimen Grids for EDXA Calibration*—TEM specimen grids prepared from dispersion of calibration minerals required for calibration of the EDXA system: crocidolite (NIST SRM 1866) and chrysotile.

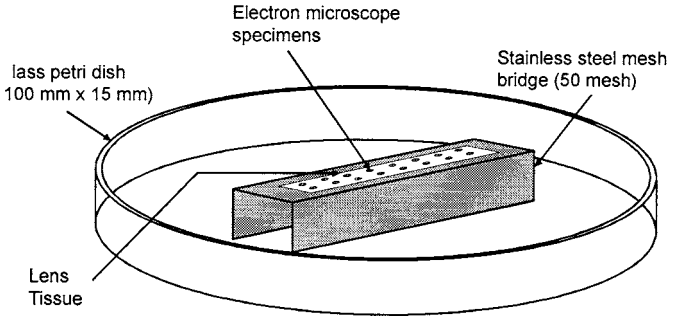


FIG. 1

7.3.2 *Energy Dispersive X-ray Analyzer*—The TEM shall be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 180 eV (FWHM) on the MnKα peak. The performance of an individual TEM-EDXA system is dependent on a number of geometrical factors. Therefore, the required performance of the TEM-EDXA system is specified in terms of the measured X-ray obtained from a fiber of small diameter, using a known electron beam diameter. Solid state X-ray detectors are least sensitive in the low energy region; therefore, measurement of sodium in crocidolite shall be the performance criterion. Irradiation of a UICC crocidolite fiber (50 nm or smaller diameter) by an electron probe (250 nm or smaller diameter), the TEM-EDXA system shall yield, under routine analytical conditions, a background-subtracted NaKα integrated peak count rate of more than 1 count per second (cps). The peak/background ratio for this performance test shall exceed 1:0.

7.3.2.1 The EDXA unit shall provide the means for subtraction of the background, identification of elemental peaks, and calculation of background-subtracted peak areas.

7.3.3 *Grating Replica*, approximately 2000 parallel lines per mm, used to calibrate the magnification of the TEM.

7.3.4 *Reference Asbestos Samples*, for preparation of reference TEM specimens of the primary asbestos minerals. The UICC or NIST mineral set is suitable for this purpose.

7.3.5 *Transmission Electron Microscope*—A TEM operating at an accelerating potential from 80 to 120 kV, with a resolution better than 1.0 nm, and a magnification range of approximately 300 to 100 000 × shall be used, with the ability to obtain a screen magnification of about 100 000 ×, for inspection of fiber morphology. This magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular. It is also required that the viewing screen of the microscope be calibrated such that the lengths and widths of fiber images down to 1–mm width can be estimated in increments of 1 mm regardless of fiber orientation. This requirement is often fulfilled through use of a fluorescent screen with calibrated gradation in the form of circles.

7.3.5.1 For Bragg angles less than 0.01 radians, the TEM shall be capable of performing ED from an area of 0.6 μm^2 or less. This performance requirement defines the minimum separation between particles at which independent ED patterns can be obtained from each particle. If ED is used, the performance of a particular instrument may normally be calculated using the following relationship:

$$A = 0.7854 \left[\frac{D}{M} + 2000 C_s \theta^3 \right]^2 \quad (1)$$

where:

A = the effective ED area, μm^2,
D = the diameter of the ED aperture, μm,
M = the magnification of the objective lens,
C_s = the spherical aberration coefficient of the objective lens, mm, and
θ = maximum required Bragg angle, radians.

7.3.5.2 It is not possible to reduce the effective ED area indefinitely by the use of progressively smaller ED apertures because there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

7.3.5.3 If zone axis ED analyses of amphiboles are to be performed, the TEM shall incorporate a goniometer stage that permits the TEM specimen to be either:

(*a*) Rotated through 360°, combined with tilting through at least +30 to -30° about an axis in the plane of the specimen; or

(*b*) Tilted through at least +30 to -30° about two perpendicular axes in the plane of the specimen.

7.3.5.4 The analysis is greatly facilitated if the goniometer permits eucentric tilting, although this is not essential. If EDXA and zone-axis ED are required on the same fiber, the goniometer shall be of a type that permits tilting of the specimen and acquisition of EDXA spectra without change of specimen holder. If the goniometer does not permit eucentric tilting, gold or other metal film must be evaporated on the sample in order that ED patterns may be accurately calibrated.

7.3.5.5 The TEM shall have an illumination and condenser lens system capable of forming an electron probe smaller than 250 nm in diameter. It is recommended that an anticontamination trap be used around the specimen.

8. Reagents

8.1 *Reagents for Sample Preparation*:

8.1.1 *1-Methyl-2-pyrrolidone*, analytical grade.

8.1.2 *1-2-diaminoethane*, analytical grade.

8.1.3 *Acetone*, analytical grade.

8.1.4 *Alcohol*, ethanol, 2-propanol, or methanol.

8.1.5 *Chloroform*, analytical grade, distilled in glass (preserved with 1 % (v/v) ethanol).

8.1.6 *Dimethyl Formamide*, analytical grade.

8.1.7 *Glacial Acetic Acid*, analytical grade.

8.1.8 *Purity of Water*—References to water shall be understood to mean reagent water as defined by Type I of Specification D 1193, or by distilled or deionized water filtered through a membrane filter of 0.22 μm maximum pore size. (**Warning—**Use the reagents in accordance with the appropriate health and safety regulations. Review their Material Safety Data Sheets before use.)

9. Procedure

9.1 Identify and document all areas to be sampled. Documentation should include:

9.1.1 General sampling site description.

9.1.2 Project or client name, address, and city/state location.

9.1.3 Sample location, which should include all information needed to locate the room and where the sample was collected. These include building, floor, room number, and room name.

NOTE 1—Some investigators include dimensions from some sort of reference (for example, 3 ft. 0 in. (0.9 m) from outside wall and 2 ft. 0 in. (0.6 m) from north wall), whereas others provide a section allowing such information to be recorded on a sample collection sheet.

9.1.4 Surface type, which should include descriptors of the surfaces in the room upon which the samples were collected. These include floor, wall, ceiling, top of light fixture, top of ceiling tile, exterior or duct, and so forth. It is sometimes useful to provide a section allowing for identification of surface sampled (for example, for a louver, whether the sample is from the top or bottom surface; for a grill, whether the sample is from the upstream or downstream side).

9.1.5 Surface material, which should describe the material from which the surface is constructed (for example, painted plaster or drywall, wood, concrete, metal, fabric, brick, resilient flooring, and so forth).

9.1.6 Surface description, which should describe the nature of the surface (for example color, texture, clean, dry, greasy, wet).

9.1.7 The area of surface wiped. It may not always be possible to collect from 100 cm^2 of surface. For example, one should indicate whether the effective surface area of a grill is discounted for the open spaces in the grill.

9.1.8 Post sampling cleanliness of surface. A visual evaluation of the cleanliness of the surface post-sampling should be made and recorded. This evaluation should not be made until the surface has dried.

9.2 Two sampling procedures are presented (see EPA 747-R-95-001). One procedures for sampling in unrestricted areas such as floors (Template Assisted Sampling Procedure). The Confined Area Sampling Procedure should only be used when the Template Assisted Sampling Procedure can not be used due to sampling location constraints. The Confined Area Sampling Procedure assumes the width of the sampling location is no larger than the dimensions of a wipe. If this is not true, then the Template Assisted Sampling Procedure is used.

9.2.1 *Template Assisted Sampling Procedure*:

9.2.1.1 If a reuseable template is used, clean template (see 7.1.6).

9.2.1.2 Determine, measure, mark, or mask area, or place template onto surface. Document the location and area (cm^2) of surface to be sampled.

9.2.1.3 A typical sampling area is 100 cm^2. Smaller or larger areas may be sampled depending on surface cleanliness.

9.2.1.4 Put on a pair of clean, powderless, rubber gloves.

9.2.1.5 Adequately moisten the wipe with a 50/50 mixture of alcohol and water. For example, 10 to 20 mL will adequately moisten a 21 by 21 cm wipe. It is recommended that a portion of the wipe be tested with the mixture if there is any doubt that the solvent may damage the wipe material.

9.2.1.6 *First Wiping, Side to Side*—Hold one edge of the wipe between the thumb and forefinger, draping the wipe over the fingers of a gloved hand. Hold fingers together, hand flat, and wipe the selected surface area, starting at either corner furthest away from the operator (referred to as a far corner), and use a slow side to side (left to right or right to left) sweeping motion. During wiping, apply even pressure to the fingertips.

9.2.1.7 At the end of the first side to side pass, turn the wipe's leading edge (portion of the wipe touching the surface) 180°. Pull the wipe path slightly close to the operator and make a second side to side pass in the reverse direction, slightly overlapping the first pass. The 180° turn is used to ensure that the wiping motion is always performed in the same direction on the wipe to maximize sample pickup. Continue to cover the sampling area within the template, using the slightly overlapping side to side passes with the 180° turns at each edge until the close corner of the template is reached. Carefully lift the sampled material into the wipe, using a slight rolling motion of the hand to capture the sample inside the wipe. Fold the wipe in half with the sample folded inside the fold.

9.2.1.8 *Second Wiping, Top to Bottom*—Using a clean side of the wipe, perform a second wiping over the sampling area within the template, starting from a far corner in the same manner used for the first wiping, except use a top to bottom sweeping of the surface. When the close corner of the template is reached, carefully lift the sampled material into the wipe, using a slight rolling motion of the hand to capture the sample inside the wipe. Fold the wipe in half again, with the sample from this second wiping folded inside the fold.

9.2.1.9 *Third Wiping, Clean Corners*—Using a clean side of the wipe, perform a third wiping around the perimeter of the sampling area within the template. Start from one edge of the template and use the same wiping technique as described in 9.2.1.8. When the interior perimeter has been wiped and the starting location reached, carefully lift the sampled material into the wipe, using a slight rolling motion of the hand to capture the sample inside the wipe. Fold the wipe in half one more time, with the sample from this third wiping folded inside the fold.

9.2.1.10 Insert the folded wipe into a sample container and seal. Label the container with sample number and sufficient information to uniquely identify the sample.

9.2.1.11 If the template is a reusable type, clean the template (see 7.1.6).

9.2.1.12 Discard gloves.

9.2.1.13 Check that all sampling information sheets are completed and that all pertinent information has been enclosed before transferring the samples to the laboratory

9.2.1.14 Collect a field wipe (see 3.2.18).

9.2.1.15 Wipe off the exterior surface of the sample containers with disposable wet towels prior to packaging for shipment.

9.2.2 *Confined Area Sampling Procedure*:

9.2.2.1 Put on a pair of clean, powderless, rubber gloves.

9.2.2.2 Adequately moisten the wipe with a 50/50 mixture of alcohol and water. (For example, 10 to 20 mL will adequately moisten a 21 by 21 cm wipe.) It is recommended that a portion of the wipe be tested with the mixture if there is any doubt that the solvent may damage the wipe material.

9.2.2.3 *First Wiping, One Direction, Side-to-Side*—Hold one edge of the wipe between the thumb and forefinger, draping the wipe over the fingers of a gloved hand. Hold fingers together, hand flat, and wipe the selected surface area. Start at either corner furthest away from the operator (referred to as a far corner), and use a slow side to side (left to right or right to left) sweeping motion. During wiping, apply even pressure to the fingertips. At the end of the first pass from one side to the other, carefully lift the sample material into the wipe, using a slight rolling motion of the hand to capture the sample inside the wipe. Fold the wipe in half with the sample folded inside the fold.

9.2.2.4 *Second Wiping, One Direction, Side-to-Side*—Using a clean side of the wipe, repeat step 9.2.2.3, using a wiping motion in the reverse direction. When the close corner of the sampling area is reached, carefully lift the sampled material into the wipe, using a slight rolling motion of the hand to

capture the sample inside the wipe. Fold the wipe in half again, with the sample from this second wiping folded inside the fold.

9.2.2.5 *Third Wiping, Clean Corners*—Using a clean side of the wipe, perform a third wiping around the interior perimeter of the sampling area. Start from the middle of one edge of an area and use the same wiping technique as described in 9.2.2.3. When the perimeter has been wiped and the starting location reached, carefully lift the sample material into the wipe, using a slight rolling motion of the hand to capture sample inside the wipe. Fold the wipe in half one more time with the sample from this third wiping folded inside the fold.

9.2.2.6 Insert the folded wipe into a sample container and seal. Label the sample container with sample number and sufficient information to uniquely identify the sample.

9.2.2.7 Discard gloves.

9.2.2.8 Using a tape measure, measure the dimension of the sampled surface with units such as inches or centimetres.

9.2.2.9 Check that all sampling information sheets are completed and that all pertinent information has been enclosed before transfer of the sample to the laboratory

9.2.2.10 Collect a field wipe.

9.2.2.11 Wipe off the exterior surface of the sample transport containers with disposable wet towels prior to packaging for shipment.

10. Sample Shipment

10.1 Ship samples to an analytical laboratory, separately packed from any bulk or air samples. The samples shall be packed in a material fiber-free material to minimize the potential for contamination.

NOTE 2—One package containing a large number of wipes moistened with a 50/50 mixture of alcohol and water may fall under regulations regarding transportation of dangerous goods. Prior to shipment, contact either International Air Transport Association (IATA) Dangerous Good Regulations for air shipment or Department of Transportation (DOT) for ground shipment.

11. Sample Suspension Preparation

11.1 Before taking sample containers into a clean preparation area, carefully wet-wipe the exterior of the containers to remove any possible contamination.

11.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and airborne asbestos sample preparation areas.

11.3 Initial specimen preparation (see 11.3.1-11.3.6) shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing surface wipe sample.

11.3.1 Transfer the wipe into a clean, wide-mouthed specimen bottle.

11.3.2 Rinse out the interior of the sample transport container with a known volume of water.

11.3.3 Pour this rinse water into the specimen bottle containing the wipe.

11.3.4 Add an additional measured volume into the labeled specimen bottle to submerge the wipe in 500 mL of water.

11.3.5 Using forceps, carefully unfold the wipe to expose all of the surfaces.

11.3.6 Adjust the pH of the suspension to 3 to 4, using a 10.0 % solution of acetic acid. Use pH paper for testing.

11.3.7 Replace the top to the specimen bottle, and lightly shake the suspension by hand for 3 s.

11.4 Place the specimen bottle in a tabletop ultrasonic bath. Maintain the water level in the sonicator at the same height as the suspension in the specimen bottle. Sonicate for 5.0 min to release particles from wipe.

11.4.1 Calibrate the ultrasonic bath as described in 21.4.2. Operate the ultrasonic bath at equilibrium temperature. After sonication, return the specimen bottle to the work surface of the HEPA hood.

12. Blank Filtration

12.1 Process at least one field wipe (see 3.2.18) along with each batch of samples to test for potential contamination during the sampling, shipping, handling, and preparation steps of the test method. Reject the sample set or take appropriate actions if relatively high fiber counts are determined.

12.2 In addition, process sample blanks that include a process blank (see 3.2.25) and filtration blank (see 3.2.19). If glass filtration units are used, prepare a filtration blank prior to each new use of the filtration unit.

12.3 The process and filtration blanks will be considered contaminated if, after analysis, they are shown to contain more than 53 asbestos structures per square millimetre of the analyzed filter. This generally corresponds to 3 or 4 asbestos structures found in 10 grid squares. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks, and prepare new samples after the source of the contamination is found.

13. Sample Filtration

13.1 Use a filtration unit for filtration of suspension aliquots.

13.2 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus. Retain the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000-mL side arm vacuum flask. Place a 5.0-µm pore size MCE (backing filter) on the support pad.

13.3 Wet the backing filter with a few millilitres of water, and place a ≤0.22-µm MCE or a ≤0.2-µm PC filter on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface require the discard of the ≤0.22-µm MCE or the ≤0.2-µm PC filter.

13.4 Once the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

NOTE 3—When using a PC filter, the filter must not be allowed to dry before filtration. PC filters are hydrophobic. A water-soluble wetting agent is applied to the surface in order to make the surface hydrophilic. Once this agent is removed and the filter allowed to dry, filtration through the PC filter is almost impossible.

13.5 If a glass filtration unit is used, place a 5-µm pore size MCE (backing filter) on the glass frit surface.

13.6 Wet the backing filter with a few mL of water, and place an MCE or PC filter (≤ 0.22-µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface.

13.7 If aliquots of the same sample are filtered in order of increasing concentration or volume, the glass filtration unit need not be washed between filtration.

13.8 After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, and then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in water.

13.9 With the flask at atmospheric pressure, add 20 mL of water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

13.10 Shake the sample suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

13.11 Insert a new pipette into the sample suspension to withdraw an aliquot from the central region of the suspension. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the aliquot from the pipette into the water in the funnel. Stir with pipette to mix thoroughly.

13.11.1 Estimate the amount of suspension to be withdrawn to produce an adequate filter preparation. A light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume.

13.11.2 To ensure that an optimally loaded filter is obtained, it is recommended that filters be prepared from varying aliquots of the suspension. A 3 to 10 % particulate coverage of the grid opening is ideal.

13.11.2.1 If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

13.11.3 If after examination in the TEM, the smallest volume measured (1.0 mL) yields an overloaded sample, perform a sample dilution.

13.11.3.1 If a sample dilution is required, repeat 13.10 before the dilution aliquot is taken. Do not resonicate the original suspension or any sample dilutions. Mix 10 mL of the sample suspension with 90 mL of water in a clean specimen bottle to obtain a 1:10 dilution. Follow good laboratory practices when performing dilutions.

13.12 Apply vacuum to the flask, and draw the suspension through the filter.

13.13 Discard the pipette.

13.14 Disassemble the filtering unit, and carefully remove the sample filter with fine forceps. Place the completed sample filter, sample surface side up into a precleaned, labeled disposable plastic petri dish or other similar container.

13.15 There are many practical methods for drying both MCE and PC filters, for example, drying filters in a plastic petri dish on a slide warmer or in a low temperature oven at 65 to 70°C for 10 to 15 min.

13.16 Prepare TEM specimens from small sections of each dried filter, using the appropriate direct transfer preparation method (see Sections 14 and 15).

14. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 4—Use of either the acetone or the dimethyl formamide (DMF)-acetic acid method is acceptable.

14.1 *Acetone Fusing Method*:

14.1.1 Process at least one filter blank with every batch of samples.

14.1.2 Remove a section from any quadrant of the sample and blank filters. Sections can be removed from the filters using either a scalpel or 7-mm cork borer. The scalpel or cork borer must be wet- wiped after each time a section is removed.

14.1.3 Place the filter section (sample side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement, or other suitable means. Label the slide with a glass scribing tool or permanent marker.

14.1.4 Prepare a fusing dish from a glass petri dish and a metal screen bridge with a pad of five to six paper filters, and place in the bottom of the petri dish (see 40 CFR 763). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish, and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

14.2 *DMF-Acetic Acid Method*:

14.2.1 Process at least one filter blank with every batch of samples.

14.2.2 Place a drop of clearing solution that consists of 35 % DMF, 15 % glacial acetic acid, and 50 % water (v/v) on a clean microscope slide. Gage the amount used so that the clearing solution just saturates the filter section.

14.2.3 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess collapsing solution by allowing an absorbent tissue to contact the liquid at the edge of the filter. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

14.3 *Plasma Etching of the Collapsed Filter*:

14.3.1 The plasma asher shall be calibrated as described in 21.4.4.

14.3.2 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher.

14.3.3 Position the slide with portions of collapsed MCE filter approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized.

14.3.4 Place the glass slide containing the collapsed filters into the plasma asher, and etch the filter using the optimum conditions and time. Admit air slowly to the chamber after etching, and remove the microscope slide.

14.3.4.1 Adjust the air admission valve of the plasma asher so that the time taken for the chamber to reach atmospheric pressure exceeds 2 min. Rapid air admission may disturb particulate matter on the surface of the etched filter.

14.4 *Carbon Coating of the Collapsed and Etched Filters*:

14.4.1 Carbon coating shall be performed with a high-vacuum coating unit, capable of less than 10^{-4} torr (13 mPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

14.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 mPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

14.5 *Preparation of the Solvent Washer (Jaffe Washer)*—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used **(5)** (see EPA 600/4-83-043). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

14.5.1 Place several pieces of lens tissue on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone or DMF until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 1 (see EPA 600/4-83-043).

14.6 Using a curved scalpel blade, excise at least 3 by 3-mm square pieces of the carbon-coated MCE filter from the glass slide. Place the square filter piece carbon-side up onto the shiny side of a TEM specimen grid. Place the whole filter/grid assembly onto the saturated lens tissue in the Jaffe washer.

14.7 Prepare three specimen grids from each sample.

14.8 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have *cleared*, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

14.9 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, labeled grid box.

14.10 Place the lid on the Jaffe washer, and allow the system to stand for several hours.

15. TEM Specimen Preparation of Polycarbonate (PC) Filter

15.1 Use a cleaned microscope slide to support representative portions of the PC filter during carbon evaporation. Apply two parallel strips of double-sided adhesive tape along the length of the slide, separated by a distance of approximately 22 mm.

15.2 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip shall contact the double-sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label next to each PC strip with the individual sample number.

15.3 PC filters do not require etching. Carbon coat the PC filter strips as discussed in 14.4. (**Warning—**Do not overheat the filter section while carbon coating.)

15.4 Prepare a Jaffe washer as described in 14.5, but fill the washer with chloroform to the level of the screen.

15.4.1 Using a clean curved scalpel blade, excise three 3 by 3 mm square filter pieces from each carbon-coated PC strip. Place the filter squares carbon side up onto the shiny side of a TEM grid. Place the whole filter/grid assembly onto the lens tissue in the Jaffe washer.

15.4.2 Prepare three specimen grids from each sample.

15.4.3 Place the lid on the Jaffe washer, and allow to stand for at least 4 h. Best results are obtained with longer times, up to 12 h.

15.5 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean labeled grid box.

15.6 *1-methyl-2-pyrrolidone Method*:

15.6.1 Prepare a Jaffe washer as described in 14.5, but fill the washer with a mixture of 20 % 1,2–diaminoethane (ethylene diamine) and 80 % 1-methyl-2-pyrrolidone to the level of the screen.

15.6.2 Using a clean curved, scalpel blade, excise three 3 by 3 mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together, and place them directly on the stainless steel mesh of the Jaffe washer (do not use lens paper) for 15 min.

15.6.3 Prepare three specimen grids from each sample.

15.6.4 Transfer the stainless steel bridge into another petri dish, and add distilled water until the meniscus contacts the underside of the mesh. Wait 15 min.

15.7 Remove mesh, and allow grids to dry before placing them in a clean, marked grid box.

16. Grid Opening Measurements

16.1 TEM grids shall have a known grid opening area. Determine this area by one of the following methods:

16.1.1 Measure at least 20 grid openings in each of 20 random 200-mesh electron microscope grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

16.1.2 Grid opening area measurements can also be made at the TEM at a suitable calibrated screen magnification such that an entire grid opening is visible within the calibration area of the screen at one time. Typically, one grid opening for each grid examined is measured. Measure grid openings in both the *X* and *Y* directions, and calculate the area.

16.1.3 Commercially available precalibrated TEM grids are also acceptable for this test method.

17. TEM Examination

17.1 *Microscope Settings*—80 to 120 kV, 15 000 to 20 000 × screen magnification for analysis.

17.2 For the analyses, analyze two grids for each sample. Select approximately half of the grid openings to be examined from each of the two grids.

17.3 *Determination of Specimen Suitability*—Use a hand lens or loupe if necessary. Carefully load the TEM grid into the specimen holder so that the carbon side will be facing down in the TEM column, with the grid bars oriented parallel/perpendicular to the length of the specimen holder. This procedure will line up the TEM grid with the *X* and *Y* translation directions of the microscope. Insert the specimen holder into the microscope.

17.3.1 Valid data cannot be obtained unless the TEM specimens meet specified quality criteria. Examine the specimen grid in the TEM at a sufficiently low magnification (300 to 1 000 ×) so that complete grid opening can be inspected. Reject the grid if:

17.3.1.1 The specimen grid has not been cleared of filter medium by the filter dissolution step. If the TEM specimen exhibits areas of undissolved filter medium, and if at least two of the three specimen grids are not cleared, perform additional solvent washing, or prepare new specimens from the filter;

17.3.1.2 The sample is over-loaded with particulate matter. If the specimen grid exhibits more than approximately 10 % obscuration on the majority of the grid openings, designate the specimen preparation as over-loaded. This filter cannot be satisfactorily analyzed because the grid is too heavily loaded with debris to allow separate examination of individual particles by ED and EDXA, and obscuration of fibers by other particulate matter may lead to under-estimation of the asbestos structure count;

17.3.1.3 The particulate loading on the specimen is not uniformly distributed from one grid opening to the next. If the particulate loading on the specimen is obviously not uniform from one grid to the next, designate the specimen as nonuniform. Satisfactory analysis of this filter may not be possible unless a large number of grid openings are examined;

17.3.1.4 The TEM grid is too heavily loaded with fibrous structures to make an accurate count. Accurate counts cannot be made if the grid has more than approximately 30 asbestos fibers per grid opening; or

17.3.1.5 More than approximately 25 % of the grid openings have broken carbon film over the whole grid opening. Since the breakage of carbon film is usually more frequent in areas of heavy deposit, counting the intact grid openings can lead to an underestimate of the structure count.

17.3.2 If the specimens are rejected because unacceptable numbers of grid openings exhibit broken carton replica, apply an additional carbon coating to the carbon-coated filter, and prepare new specimen grids. A thicker carbon film can often support the larger particles. However, too thick a carbon film will reduce image contrast and have an adverse effect on the analyst's ability to discern weak diffraction patterns from thin fibers.

17.4 *Data Recording Rules*:

17.4.1 Observe and record the orientation of the grid at 80 to 150 × on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid openings must be recorded.

17.4.2 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 3.2.

17.4.3 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in 40 CFR 763. Record the length and width measurements for each identified Asbestos Hazard Emergency Response Act (AHERA) structure.

17.4.4 Record NSD (No Structures Detected) when no AHERA structures are detected in the grid opening.

17.4.5 Identify structures classified as chrysotile by either ED or EDXA, and record on a count sheet. Verify at least one out of every ten chrysotile structures by either ED or EDXA.

17.4.6 Structures classified as amphiboles by EDXA and ED are recorded on the count sheet. For more information on identification, see Ref. (**5**) or EPA 600/4-83-043.

17.4.7 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. If the X-ray spectrum is stored, record the file and disk number on the count sheet. If a hard copy is generated, retain the hard copy with the count sheets.

17.5 *Counting Rules*:

17.5.1 At a screen magnification of between 15 000 and 20 000 ×, evaluate the grids for the most concentrated sample loading. Reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample unit a set of grids are obtained that have less than 30 asbestos structures per grid opening.

17.5.2 *Analytical Sensitivity*—An analytical sensitivity of approximately 260 asbestos structures per square centimetre

(calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed or decreasing the size of the final filter. Occasionally, due to high particle loading or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply (see 17.6).

17.6 *Stopping Rules*:

17.6.1 A minimum of four grid openings shall be analyzed for each sample.

17.6.2 The nominal number of grid openings to be analyzed is the number required to achieve an analytical sensitivity of 260 asbestos structures per square centimetre. If, due to dilution or any other reason, the analytical sensitivity is not achievable by analyzing ten grid openings, stop at the grid opening that contains the hundredth asbestos structure, or completion of the tenth grid opening, whichever comes first.

17.7 After analysis, remove the grids from the TEM and replace them in the appropriate labeled grid box.

18. Sample Storage

18.1 The washed-out sample containers can be discarded after use.

18.2 Sample grids and unused filter section shall be stored for a minimum of one year.

19. Report

19.1 Report the following information for each sample analyzed:

19.1.1 Concentration of asbestos structures per square centimetre of area wiped,

19.1.2 Analytical sensitivity,

19.1.3 Detection limit,

19.1.4 Types of asbestos present,

19.1.5 Number of asbestos structures counted,

19.1.6 Effective filtration area of filter through which the suspensions were drawn (mm^2),

19.1.7 Average area of the TEM grip opening (mm^2),

19.1.8 Number of grid openings examined,

19.1.9 Initial volume of water introduced into specimen bottle containing wipe, see 11.3,

19.1.10 Volume of suspension filtered,

19.1.11 Area of surface wiped,

19.1.12 Listing of size data for each structure counted, and

19.1.13 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

19.2 Determine the amount of asbestos in any accepted sample, using the following formula:

$$\frac{EFA\ IV\ \#STR}{GO\ GOA\ V\ SA} = \text{Asbestos Structures/cm}^2 \qquad (2)$$

where:

#STR = number of asbestos structures counted,
EFA = effective filtration area of filter through which the suspension were drawn, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SA = area of surface wiped (cm^2),
V = volume of suspension filtered in 10.8, mL, and
IV = initial volume, mL. Measured volume of water introduced into the specimen bottle containing the wipe (see 10.3.4).

20. Quality Control/Quality Assurance

20.1 In general, the laboratory's quality control checks are used to verify that a system is performing in accordance with specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. At this time, due to the difficulties in prepared known quantitative asbestos samples, the accuracy of this test method cannot be determined. However, routine quality control testing focusing on reanalysis of samples is to be performed.

20.1.1 Reanalyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed, not including blanks). The reanalysis shall consist of a second specimen grid preparation.

20.2 In addition, quality assurance programs shall follow the criteria shown in 40 CFR 763 and in Ref. (**6**). These documents describe sample custody, sample preparation, and blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

21. Calibrations

21.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory in accordance with the laboratory's quality assurance program.

21.2 Record calibrations in a logbook, along with calibration dates and data.

21.3 Frequency of calibration will depend on the service history of the particular instrument/equipment.

21.4 *Calibration List for Instruments Follows*:

21.4.1 *TEM*:

21.4.1.1 Check the alignment and systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

21.4.1.2 Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

21.4.1.3 Calibrate the camera length of the TEM in ED operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only on or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown *d*-spacings of most interest in asbestos analysis are those that lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that

particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

21.4.1.4 Perform magnification calibration at the fluorescent screen. This calibration shall be performed at the magnification used for structure counting. Calibration is performed with a grating replica.

(a) Define a field of view on the fluorescent screen. The field of view shall be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) The analyst shall ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(c) For instruments that incorporate an eucentric tilting specimen stage, all specimens and the grating replica shall be placed at the eucentric position.

21.4.1.5 The dimension of the smallest spot size routinely used for EDXA analysis shall be measured.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000 ×. An exposure time of 1 s is usually adequate.

(b) The measured spot size shall be less than or equal to 250 nm.

21.4.2 *EDXA*:

21.4.2.1 The EDXA detector resolution (at the MnKα peak) and energy calibration shall be determined and monitored over time.

(a) Calibrate the EDXA using at least two peaks between 0.7 and 10 keV. One peak should be from the low end (0.7 to 2 keV) and the other peak form the high end (7 to 10 keV).

(b) Compare the X-ray energy versus the channel number, and adjust equipment if reading is not within ±10 eV.

21.4.2.2 Collect a standard EDXA of crocidolite (NIST SRM 1866). The elemental analysis of the crocidolite shall resolve the Na peak.

21.4.2.3 Collect a standard EDXA of chrysotile (NIST SRM 1866). The elemental analysis of chrysotile shall resolve both Si and Mg from a single chrysotile fiber.

21.4.3 *Ultrasonic Bath Calibration*:

21.4.3.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for sample suspension dispersion. Operate the bath until the water reaches the equilibrium temperature.

21.4.3.2 Place 100 mL of water (at approximately 20°C) in a 200 mL glass sample container, and record the water temperature.

21.4.3.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record the water temperature.

21.4.3.4 Place 100 mL of water (approximately 20°C) in another 200 mL glass sample container, and record the water temperature.

21.4.3.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record the water temperature.

21.4.3.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185\ S\ D\ \frac{(q_1 - q_2)}{t} \quad (3)$$

where:

4.185 = joules/calorie,
R = energy deposition, W/mL,
q_1 = temperature rise with the ultrasonic bath not operating °C
q_2 = temperature rise with the ultrasonic bath operating, °C
t = time, s (see 21.4.3.3),
S = specific heat of the liquid in the glass sample container (0.2389 J/g), and
D = density of the liquid in the glass sample container (1.0 g/cm^3).

21.4.3.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range from 0.08 to 0.12 MW/m^3, as defined by this procedure.

21.4.4 *Plasma Asher Calibration*:

21.4.4.1 Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that shall be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, determine a calibration curve for the weight versus etching time of collapsed MCE filters.

(a) Place a microscope slide containing a collapsed, 25-mm diameter MCE filter into the center of the asher chamber.

(b) Closes the chamber and evacuate to a pressure of 40 Pa, while admitting oxygen to the chamber at a rate from 8 to 20 mL/min. Adjust the tuning of the system so that the intensity of the plasma is maximized.

(c) Measure the time required for complete oxidation of the filter.

(d) Determine the operating parameters that result in complete oxidation of the filter in a period of 15 min. Use these operating parameters to etch a preweighed, collapsed filter for a period of 8 min. Weigh the filter after etching.

(e) Adjust the parameters and retest until a 1 to 10 % weight loss is achieved.

(f) The AHERA (see 40 CFR 763) method specifies that a MCE filter is to be etched by 10 %. However, if this amount generates a texture in the replica that affects structure counting, it is permissible to etch by less than this amount **(6)**. The final acceptance of the etched filter is dependent on its appearance in the TEM.

22. Precision and Bias

22.1 *Precision*—To be determined.

22.2 *Bias*—To be determined (see Guide D 3670).

23. Keywords

23.1 asbestos; surface sampling; TEM; wipe sampling

APPENDIX

(Nonmandatory Information)

X1. SAMPLE ANALYSIS

X1.1 See Fig. X1.1 for the analysis work sheet.

X1.2 See Fig. X1.2 for the TEM count sheet.

SAMPLE ANALYSIS

Client:		Accelerating Voltage:	
Sample ID:		Indicated Mag:	KX
Job Number:		Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:	/	Filter Type:	
Grid Accepted, 600X:	Yes No	Filter Size:	
Percent Loading:	%	Filter Pore Size (µm):	
Grid Box #1:		Grid Opening:	1) µm x µm
			2) µm x µm

Analyst: _____

Reviewer: _____

Calculation Data:

Effective Filter Area (mm^2):	(EFA)	
Number of Grid Openings Counted:	(GO)	
Average Grid Opening Area (mm^2):	(GOA)	
Volume of sample Filtered (ml):	(V)	
Surface Area Sampled (cm^2):	(SA)	
Initial Volume (ml)	(IV)	
Number of Asbestos Structures Counted:	(#STR)	

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES PER CM^2 OF SURFACE WIPED:

$$\frac{(EFA)(IV)(\#STR)}{(GO)(GOA)(V)(SA)} = \text{Asbestos Structures per } cm^2$$

Results for Total Asbestos Structures: (Structures per cm^2)

FIG. X1.1 Analysis Work Sheet

Job Number: _____

Structure #	Grid Opening#	Type	Structure	Length (μm)	Width (μm)	Confirmation		
						Morph	ED	EDXA

Note: Keys to Abbreviations Used in Figure:

Type:			**Structure:**			**Others:**		
C	=	Chrysotile	F	=	Fiber	NSD	=	No Structures Detected
AM	=	Amosite	B	=	Bundle	Morph	=	Morphology
CR	=	Crocidolite	C	=	Cluster	ED	=	Electron Diffraction
AC	=	Actinolite	M	=	Matrix	EDXA	=	Energy Dispersive X-Ray Analysis
TR	=	Tremolite						
AN	=	Anthophyllite						
N	=	Non Asbestos						

FIG. X1.2 TEM Count Sheet

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INTERNATIONAL STANDARD

ISO 10312

First edition
1995-05-01

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

Air ambiant — Détermination des fibres d'amiante — Méthode de microscopie électronique à transmission directe

Reference number
ISO 10312:1995(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10312 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Annexes A, B, C, D, E and F form an integral part of this International Standard. Annexes G, H and J are for information only.

Introduction

This International Standard is applicable to the determination of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for detailed evaluation of any atmosphere in which asbestos structures are likely to be present. Because the best available medical evidence indicates that the numerical fibre concentration and the fibre sizes are the relevant parameters for evaluation of the inhalation hazards, a fibre counting technique is the only logical approach. Most fibres in ambient atmospheres are not asbestos, and therefore there is a requirement for fibres to be identified. Many airborne asbestos fibres in ambient atmospheres have diameters below the resolution limit of the optical microscope. This International Standard is based on transmission electron microscopy, which has adequate resolution to allow detection of small fibres and is currently the only technique capable of unequivocal identification of the majority of individual fibres of asbestos. Asbestos is often found, not as single fibres, but as very complex, aggregated structures which may or may not be also aggregated with other particles. The fibres found suspended in an ambient atmosphere can often be identified unequivocally, if a sufficient measurement effort is expended. However, if each fibre were to be identified in this way, the analysis would become prohibitively expensive. Because of instrumental deficiencies or because of the nature of the particulate, some fibres cannot be positively identified as asbestos, even though the measurements all indicate that they could be asbestos. Subjective factors therefore contribute to this measurement, and consequently a very precise definition of the procedure for identification and enumeration of asbestos fibres is required. The method specified in this International Standard is designed to provide the best description possible of the nature, numerical concentration, and sizes of asbestos-containing particles found in an air sample. This International Standard is necessarily complex, because the instrumental techniques used are complex, and also because a very detailed and logical procedure must be specified to reduce the subjective aspects of the measurement. The method of data recording specified in this International Standard is designed to allow re-evaluation of the structure counting data as new medical evidence becomes available. All of the feasible specimen preparation techniques result in some modification of the airborne particulate. Even the collection of particles from a three-dimensional airborne dispersion onto a two-dimensional filter surface can be considered a modification of the particulate, and some of the particles in most samples are modified by the specimen preparation procedures. However, the procedures specified in this International Standard are designed to minimize the disturbance of the collected particulate material, and the effect of those disturbances which do occur can be evaluated.

This International Standard describes the method of analysis for a single air filter. However, one of the largest potential errors in characterizing asbestos in ambient atmospheres is associated with the variability between filter samples. For this reason, it is necessary to design a replicate sampling scheme in order to determine this International Standard's accuracy and precision.

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

1 Scope

1.1 Substance determined

This International Standard specifies a reference method using transmission electron microscopy for the determination of the concentration of asbestos structures in ambient atmospheres and includes measurement of the lengths, widths and aspect ratios of the asbestos structures. The method allows determination of the type(s) of asbestos fibres present. The method cannot discriminate between individual fibres of the asbestos and non-asbestos analogues of the same amphibole mineral.

1.2 Type of sample

The method is defined for polycarbonate capillary-pore filters or cellulose ester (either mixed esters of cellulose or cellulose nitrate) filters through which a known volume of air has been drawn. The method is suitable for determination of asbestos in both exterior and building atmospheres.

1.3 Measuring range

The range of concentration which can be determined is 50 structures/mm^2 to 7 000 structures/mm^2 on the filter. The air concentrations represented by these values are a function of the volume of air sampled. There is no lower limit to the dimensions of asbestos fibres which can be detected. In practice, microscopists vary in their ability to detect very small asbestos fibres. Therefore, a minimum length of 0,5 µm has been defined as the shortest fibre to be incorporated in the reported results.

1.4 Limit of detection

The limit of detection theoretically can be lowered indefinitely by filtration of progressively larger volumes of air and by extending the examination of the specimens in the electron microscope. In practice, the lowest achievable limit of detection for a particular area of TEM specimen examined is controlled by the total suspended particulate concentration.

For total suspended particulate concentrations of approximately 10 µg/m^3, corresponding to clean, rural atmospheres, and assuming filtration of 4 000 litres of air, an analytical sensitivity of 0,5 structure/l can be obtained, equivalent to a limit of detection of 1,8 structure/l, if an area of 0,195 mm^2 of the TEM specimens is examined. If higher total suspended particulate concentrations are present, the volume of air filtered must be reduced in order to maintain an acceptable particulate loading on the filter, leading to a proportionate increase in the analytical sensitivity.

Where this is the case, lower limits of detection can be achieved by increasing the area of the TEM specimens that is examined. In order to achieve lower limits of detection for fibres and bundles longer than 5 µm, and for PCM equivalent fibres, lower magnifications are specified which permit more rapid examination of larger areas of the TEM specimens when the examination is limited to these dimensions of fibre. The direct analytical method cannot be used if the general particulate loading of the sample collection filter exceeds approximately 10 µg/cm^2 of filter surface, which corresponds to approximately 10 % coverage of the collection filter by particulate. If the total suspended particulate is largely organic material, the limit of detection can be lowered significantly by using an indirect preparation method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4225:1994, *Air quality — General aspects — Vocabulary.*

ISO 4226:1993, *Air quality — General aspects — Units of measurement.*

ISO Standard Handbook No. 2:1993, *Quantities and units.*

ISO Standard Handbook No. 3:1989, *Statistical Methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply (see also ISO 4225).

3.1 acicular: The shape of an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like.

3.2 amphibole: A group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, with the nominal formula:

$$A_0 \text{ or } {}_1B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where

A = K, Na

B = Fe^{2+}, Mn, Mg, Ca, Na

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+}

T = Si, Al, Cr, Fe^{3+}, Ti

In some varieties of amphibole, these elements can be partially substituted by Li, Pb or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

3.3 amphibole asbestos: Amphibole in an asbestiform habit.

3.4 analytical sensitivity: The calculated airborne asbestos structure concentration in asbestos structures/litre, equivalent to counting of one asbestos structure in the analysis. The method in this International Standard does not specify an analytical sensitivity.

3.5 asbestiform: A specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility.

3.6 asbestos: A term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, strong fibres when crushed or processed. The Chemical Abstracts Service Registry Numbers of the most common asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grünerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

3.7 asbestos structure: A term applied to any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles.

3.8 aspect ratio: The ratio of length to width of a particle.

3.9 blank: A structure count made on TEM specimens prepared from an unused filter, to determine the background measurement.

3.10 camera length: The equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action.

3.11 chrysotile: A fibrous mineral of the serpentine group which has the nominal composition

$$Mg_3Si_2O_5(OH)_4$$

Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

3.12 cleavage: The breaking of a mineral along one of its crystallographic directions.

3.13 cleavage fragment: A fragment of a crystal that is bounded by cleavage faces.

3.14 cluster: A structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping.

3.15 d-spacing: The distance between identical adjacent and parallel planes of atoms in a crystal.

3.16 electron diffraction: A technique in electron microscopy by which the crystal structure of a specimen is examined.

3.17 electron scattering power: The extent to which a thin layer of substance scatters electrons from their original directions.

3.18 energy dispersive X-ray analysis: Measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyser system.

3.19 eucentric: The condition when the area of interest of an object is placed on a tilting axis at the intersection of the electron beam with that axis and is in the plane of focus.

3.20 field blank: A filter cassette which has been taken to the sampling site, opened, and then closed. Such a filter is used to determine the background structure count for the measurement.

3.21 fibril: A single fibre of asbestos, which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.22 fibre: An elongated particle which has parallel or stepped sides. For the purposes of this International Standard, a fibre is defined to have an aspect ratio equal to or greater than 5:1 and a minimum length of 0,5 µm.

3.23 fibre bundle: A structure composed of parallel, smaller diameter fibres attached along their lengths. A fibre bundle may exhibit diverging fibres at one or both ends.

3.24 fibrous structure: A fibre, or connected grouping of fibres, with or without other particles.

3.25 habit: The characteristic crystal growth form, (or combination of these forms), of a mineral, including characteristic irregularities.

3.26 limit of detection: The calculated airborne asbestos structure concentration in structures per litre, equivalent to counting 2,99 asbestos structures in the analysis.

3.27 matrix: A structure in which one or more fibres, or fibre bundles, touch, are attached to, or partially concealed by, a single particle or connected group of nonfibrous particles.

3.28 Miller index: A set of either three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes.

3.29 PCM equivalent fibre: A fibre of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.30 PCM equivalent structure: A fibrous structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.31 primary structure: A fibrous structure that is a separate entity in the TEM image.

3.32 replication: A procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

3.33 selected area electron diffraction: A technique in electron microscopy in which the crystal structure of a small area of a sample is examined.

3.34 serpentine: A group of common rock-forming minerals having the nominal formula

$$Mg_3Si_2O_5(OH)_4$$

3.35 structure: A single fibre, fibre bundle, cluster or matrix.

3.36 twinning: The occurrence of crystals of the same species joined together at a particular mutual orientation, such that the relative orientations are related by a definite law.

3.37 unopened fibre: An asbestos fibre bundle of large diameter which has not been separated into its constituent fibrils or fibres.

3.38 zone-axis: The line or crystallographic direction through the centre of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone.

4 Principle

A sample of airborne particulate is collected by drawing a measured volume of air through either a

capillary-pore polycarbonate membrane filter of maximum pore size 0,4 µm or a cellulose ester (either mixed esters of cellulose or cellulose nitrate) membrane filter of maximum pore size 0,45 µm by means of a battery-powered or mains-powered pump. TEM specimens are prepared from polycarbonate filters by applying a thin film of carbon to the filter surface by vacuum evaporation. Small areas are cut from the carbon-coated filter, supported on TEM specimen grids, and the filter medium is dissolved away by a solvent extraction procedure. This procedure leaves a thin film of carbon which bridges the openings in the TEM specimen grid, and which supports each particle from the original filter in its original position. Cellulose ester filters are chemically treated to collapse the pore structure of the filter, and the surface of the collapsed filter is then etched in an oxygen plasma to ensure that all particles are exposed. A thin film of carbon is evaporated onto the filter surface and small areas are cut from the filter. These sections are supported on TEM specimen grids and the filter medium is dissolved away by a solvent extraction procedure.

The TEM specimen grids from either preparation method are examined at both low and high magnifications to check that they are suitable for analysis before carrying out a quantitative structure count on randomly-selected grid openings. In the TEM analysis, electron diffraction (ED) is used to examine the crystal structure of a fibre, and its elemental composition is determined by energy dispersive X-ray analysis (EDXA). For a number of reasons, it is not possible to identify each fibre unequivocally, and fibres are classified according to the techniques which have been used to identify them. A simple code is used to record, for each fibre, the manner in which it was classified. The fibre classification procedure is based on successive inspection of the morphology, the electron diffraction pattern for a selected area, and the qualitative and quantitative energy dispersive X-ray analyses. Confirmation of the identification of chrysotile is done only by quantitative ED, and confirmation of amphibole is done only by quantitative EDXA and quantitative zone axis ED.

In addition to isolated fibres, ambient air samples often contain more complex aggregates of fibres, with or without other particles. Some particles are composites of asbestos fibres with other materials. Individual fibres and structures that are more complex are referred to as "asbestos structures". A coding system is used to record the type of fibrous structure, and to provide the optimum description of each of these complex structures. The two codes remove the requirement to interpret the structure counting data from the microscopist, and allow this evaluation to be made later without the requirement for re-examination of the TEM specimens. Several levels of analysis are specified, the higher levels providing more rigorous approach to the identification of fibres. The procedure permits a minimum required fibre identification criterion to be defined on the basis of previous knowledge, or lack of it, about the particular sample. Attempts are then made to achieve this minimum criterion for each fibre, and the degree of success is recorded for each fibre. The lengths and widths of all classified structures and fibres are recorded. The number of asbestos structures found on a known area of the microscope sample, together with the equivalent volume of air filtered through this area, is used to calculate the airborne concentration in asbestos structures/litre of air.

5 Symbols of units and abbreviations

5.1 *Symbols of units* (see also ISO 4226 and ISO No. 2)

eV = electron volt

kV = kilovolt

l/min = litres per minute

µg = microgram (10^{-6} gram)

µm = micrometre (10^{-6} metre)

nm = nanometre (10^{-9} metre)

W = watt

5.2 Abbreviations

DMF	Dimethylformamide
DE	Electron diffraction
EDXA	Energy dispersive X-ray analysis
FWHM	Full width, half maximum
HEPA	High efficiency particle absolute
MEC	Mixed esters of cellulose
PC	Polycarbonate
PCM	Phase contrast optical microscopy
SAED	Selected area electron diffraction
SEM	Scanning electron microscope
STEM	Scanning transmission electron microscop
TEM	Transmission electron microscope

UICC Union Internationale Contre le Cancer

6 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water (6.1).

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

6.1 water, fibre-free.

A supply of freshly distilled, fibre-free water, or another source of fibre-free, pyrogen-free water shall be used.

6.2 Chloroform, analytical grade, distilled in glass, preserved with 1 % (*V/V*) ethanol.

6.3 1-Methyl-2-pyrrolidone.

6.4 Dimethylformamide.

6.5 Glacial acetic acid.

6.6 Acetone.

7 Apparatus

7.1 Air sampling — Equipment and consumable supplies

7.1.1 Filter cassette

Field monitors, comprising 25 mm to 50 mm diameter three-piece cassettes, with cowls which project less than 2 cm in front of the filter surface shall be used for sample collection. The cassette shall be loaded with either a capillary pore polycarbonate filter of maximum pore size 0,4 µm or an MEC or cellulose nitrate filter of maximum pore size 0,45 µm. Either type of filter shall be backed by a 5 µm pore size MEC or cellulose nitrate filter, and supported by a cellulose back-up pad. When the filters are in position, an elastic cellulose band or adhesive tape shall be applied to prevent air leakage. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly, so that significant air leakage around the filter cannot occur.

Representative filters from the filter lot shall be analysed as specified in 9.7 for the presence of asbestos structures before any are used for air sample collection.

7.1.2 Sampling pump

The sampling pump shall be capable of a flow-rate sufficient to achieve the desired analytical sensitivity. The face velocity through the filter shall be between 4,0 cm/s and 25,0 cm/s. The sampling pump used shall provide a non-fluctuating airflow through the filter, and shall maintain the initial volume flow-rate to within ± 10 % throughout the sampling period. A constant flow or critical orifice controlled pump meets these requirements. Flexible tubing shall be used to connect the filter cassette to the sampling pump. A means for calibration of the flow-rate of each pump is also required.

7.1.3 Stand

A stand shall be used to hold the filter cassette at the desired height for sampling, and shall be isolated from the vibrations of the pump (7.1.2).

7.1.4 Variable area flowmeter

A calibrated variable are a flowmeter with a range of approximately 1 l/min to 10 l/min is required for calibration of the air sampling system.

The variable area flowmeter shall be cleaned before use to avoid transfer of asbestos contamination from the flowmeter to the sample being collected.

7.2 Specimen preparation laboratory

Asbestos, particularly chrysotile, is present in varying quantities in many laboratory reagents. Many building materials also contain significant amounts of asbestos or other mineral fibres which may interfere with the analysis if they are inadvertently introduced during preparation of specimens. It is most important to ensure that, during preparation, contamination of TEM specimens by any extraneous asbestos fibres is minimized. All specimen preparation steps shall therefore be performed in an environment where contamination of the sample is minimized. The primary requirement of the sample preparation laboratory is that a blank determination shall yield a result which will meet the requirements specified in 9.7. A minimum facility considered suitable for preparation of TEM specimens is a laminar flow hood with positive pressure. However, it has been established that work practices in specimen preparation appear to be more important than the tape of clean handling facilities in use. Preparation of samples shall be carried out only after acceptable blank values have been demonstrated.

NOTE 1 It is recommended that activities involving manipulation of bulk asbestos samples not be performed in the

same area as TEM specimen preparation, because of the possibilities of contaminating the TEM specimens.

7.3 Equipment for analysis

7.3.1 Transmission electron microscope

A TEM operating at an accelerating potential of 80 kV to 120 kV, with a resolution better than 1,0 nm, and a magnification range of approximately × 300 to × 100 000 shall be used. The ability to obtain a direct screen magnification of about × 100 000 is necessary for inspection of fibre morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. It is also required that the viewing screen of the microscope be calibrated such that the lengths and widths of fibre images down to 1 mm width can be measured in increments of 1 mm, regardless of image orientation. This requirement is often fulfilled through the use of a fluorescent screen with calibrated gradations in the form of circles, as shown in figure 1.

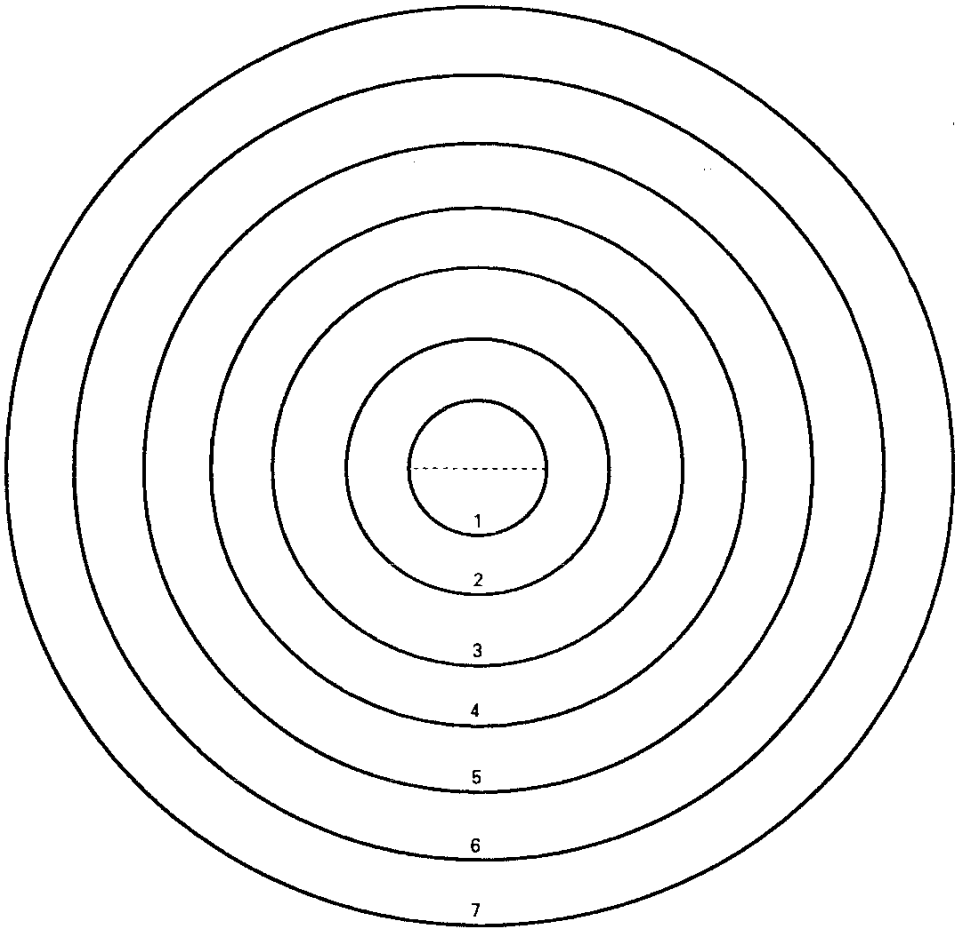


Figure 1 — Example of calibration markings on TEM viewing screen

For Bragg angles less than 0,01 rad, the TEM shall be capable of performing ED from an area of 0,6 μm^2 or less, selected from an in-focus image at a screen magnification of × 20 000. This performance requirement defines the minimum separation between particles at which independent ED patterns can be obtained from each particle. If SAED is used, the performance of a particular instrument may normally be calculated using the following equation

$$A = 0{,}785\ 4 \times \left(\frac{D}{M} + 2\ 000 C_s \theta^3 \right)^2$$

where

A is the effective SAED area, in square micrometres;

D is the diameter, in micrometres, of the SAED aperture;

M is the magnification of the objective lens;

C_s is the spherical aberration coefficient, in millimetres, of the objective lens;

θ is the maximum required Bragg angle, in radians.

It is not possible to reduce the effective SAED area indefinitely by the use of progressively smaller SAED apertures, because there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

If zone-axis ED analyses are to be performed, the TEM shall incorporate a goniometer stage which permits the TEM specimen to be either

a) rotated through 360°, combined with tilting through at least + 30° to − 30° about an axis in the plane of the specimen;

b) tilted through at least + 30° to − 30° about two perpendicular axes in the plane of the specimen.

The analysis is greatly facilitated if the goniometer permits eucentric tilting, although this is not essential. If EDXA and zone-axis ED are required on the same fibre, the goniometer shall be of a type which permits tilting of the specimen and acquisition of EDXA spectra without changing the specimen holder.

The TEM shall have an illumination and condenser lens system capable of forming an electron probe of diameter less than 250 nm.

NOTE 2 Use of an anti-contamination trap around the specimen is recommended if the required instrumental performance is to be obtained.

7.3.2 Energy dispersive X-ray analyser

The TEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 180 eV (FWHM) on the MnKα. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid state X-ray detectors are least sensitive in the low energy region, and so measurement of sodium in crocidolite shall be the performance criterion. The combination of electron microscope and X-ray analyser shall yield, under routine analytical conditions, a background-subtracted NaKα integrated peak count rate of more than 1 count per second (cps) from a fibre of UICC crocidolite, 50 nm in diameter or smaller, when irradiated by an electron probe of 250 nm diameter or smaller at an accelerating potential of 80 kV. The peak/background ratio for this performance test shall exceed 1,0.

The EDXA unit shall provide the means for subtraction of the background, identification of elemental peaks, and calculation of background-subtracted peak areas.

7.3.3 Computer

Many repetitive numerical calculations are necessary, and these may be performed conveniently by relatively simple computer programmes. For analyses of zone-axis ED pattern measurements, a computer with adequate memory is required to accommodate the more complex programmes involved.

7.3.4 Plasma asher

For preparation of TEM specimens from MEC filters, a plasma asher, with a radio frequency power rating of 50 W or higher, shall be used to etch the surface of collapsed MEC filters. The asher shall be supplied with a controlled oxygen flow, and shall be modified, if necessary, to provide a valve to control the speed of air admission so that rapid air admission does not disturb particulates from the surface of the filter after the etching step.

NOTE 3 It is recommended that filters be fitted to the oxygen supply and the air admission line.

7.3.5 Vacuum coating unit

A vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon on the membrane filters. A sample

holder is required which will allow a glass microscope slide to be continuously rotated during the coating procedure.

NOTE 4 A mechanism which also allows the rotating slide to be tilted through an angle of approximately 45° during the coating procedure is recommended. A liquid nitrogen cold trap above the diffusion pump may be used to minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum coating unit may also be used for deposition of the thin film of gold, or other calibration material, when it is required on TEM specimens as an internal calibration of ED patterns.

7.3.6 Sputter coater

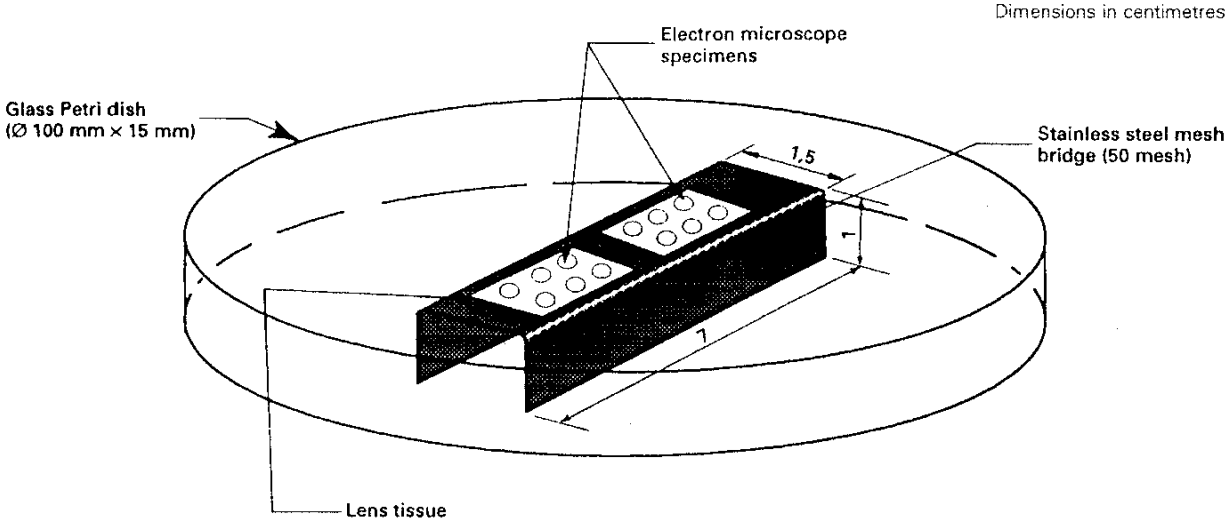
A sputter coater with a gold target may be used for deposition of gold onto TEM specimens as an integral calibration of ED patterns. Other calibration materials are acceptable. Experience has shown that a sputter coater allows better control of the thickness of the calibration material.

7.3.7 Solvent washer (Jaffe washer)

The purpose of the Jaffe washer is to allow dissolution of the filter polymer while leaving an intact evaporated carbon film supporting the fibres and other particles from the filter surface. One design of a washer which has been found satisfactory for various solvents and filter media is shown in figure 2. In general, either chloroform or 1-methyl-2-pyrrolidone has been used for dissolving polycarbonate filters and dimethylformamide or acetone has been used for dissolving MEC or cellulose nitrate filters. The higher evaporation rates of chloroform and acetone require that a reservoir of 10 ml to 50 ml of solvent be used, which may need replenishment during the procedure. Dimethylformamide and 1-methyl-2-pyrrolidone have lower vapour pressures and much smaller volumes of solvent may be used. It is recommended that all washers be used in a fume hood, and when specimens are not being inserted or removed, the Petri dish lid shall be in place during the solvent dissolution. The washer shall be cleaned before it is used for each batch of specimens.

7.3.8 Condensation washer

For more rapid dissolution of the filter polymer, or if difficulties are experienced in dissolving the filter polymer, use a condensation washer, consisting of a flask, condenser and cold finger assembly, with a heating mantle and means for controlling the temperature. A suitable assembly is shown in figure 3, using either acetone or chloroform as the solvent, depending on the type of filter.



NOTE — Solvent is added until the meniscus contacts the underside of the stainless steel mesh bridge.

Figure 2 — Example of design of solvent washer (Jaffe washer)

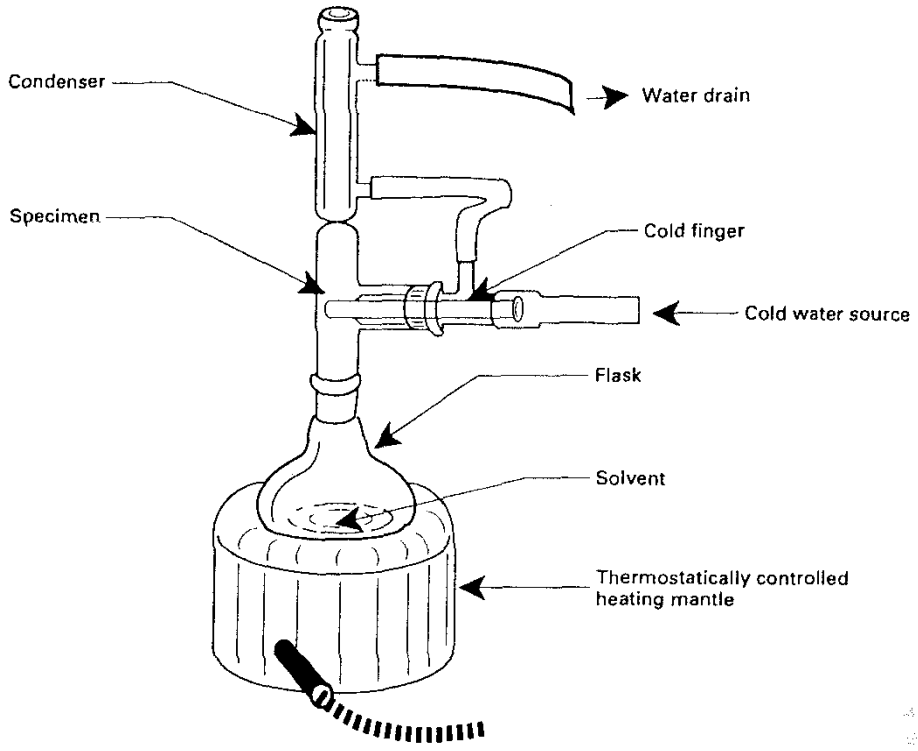


Figure 3 — Example of design of condensation washer

7.3.9 Slide warmer or oven

Use either a slide warmer or an oven for heating slides during the preparation of TEM specimens from MEC or cellulose nitrate filters. It is required to maintain a temperature of 65 °C to 70 °C.

7.3.10 Ultrasonic bath

An ultrasonic bath is necessary for cleaning the apparatus used for TEM specimen preparation.

7.3.11 Carbon grating replica

A carbon grating replica with about 2 000 parallel lines per millimetre shall be used to calibrate the magnification of the TEM.

7.3.12 Calibration specimen grids for EDXA

TEM specimen grids prepared from dispersions of calibration minerals are required for calibration of the EDXA system. Some suitable calibration minerals are riebeckite, chrysotile, halloysite, phlogopite, wollastonite and bustamite. The mineral used for calibration of the EDXA system for sodium shall be prepared using a gold TEM grid.

7.3.13 Carbon rod sharpener

The use of necked carbon rods, or equivalent, allows the carbon to be evaporated onto the filters with a minimum of heating.

7.3.14 Disposable tip micropipettes

A disposable tip micropipette, capable of transferring a volume of approximately 30 µl, is necessary for the preparation of TEM specimen grids from MEC filters.

7.4 Consumable supplies

7.4.1 Copper electron microscope grids

Copper TEM grids with 200 mesh are recommended. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.2 Gold electron microscope grids

Gold TEM grids with 200 mesh are recommended to mount TEM specimens when sodium measurements are required in the fibre identification procedure. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.3 Carbon rod electrodes

Spectrochemically pure carbon rods, shall be used in the vacuum evaporator (7.3.5) during carbon coating of filters.

7.4.4 Routine electron microscopy tools and supplies

Fine-point tweezers, scalpel holders and blades, microscope slides, double-coated adhesive tape, lens tissue, gold wire, tungsten filaments and other routine supplies are required.

7.4.5 Reference asbestos samples

Asbestos samples, shall be for preparation of reference TEM specimens of the primary asbestos minerals. The UICC set of minerals is suitable for this purpose.

8 Air sample collection

The desired analytical sensitivity is a parameter that shall be established for the analysis prior to sample collection. It is defined as the structure concentration corresponding to the detection of one structure in the analysis. For direct transfer methods of TEM specimen preparation, the analytical sensitivity is a function of the volume of air sampled, the active area of the collection filter, and the area of the TEM specimen over which structures are counted. If total airborne dust levels are high, it may be necessary to terminate sampling before the required volume has been sampled. If this happens, the analytical sensitivity required can be achieved only by counting structures on more grid openings, or by selective concentration of asbestos structures using an indirect TEM specimen preparation technique. Select the sampling rate and the period of sampling to yield the required analytical sensitivity, as detailed in table 1. Before air samples are collected, unused filters shall be analysed as described in 9.7 to determine the mean asbestos structure count for blank filters.

Air samples shall be collected using filter cassettes (7.1.1). During sampling, the cassette shall be supported on a stand (7.1.3) which is isolated from the vibrations of the pump (7.1.2). The cassette shall be held facing vertically downwards at a height of approximately 1,5 m to 2,0 m above ground/floor level, and shall be connected to the pump with a flexible tube.

Measure the sampling flow-rate at the front end of the cassette, both at the beginning and end of the sampling period, using a calibrated variable area flowmeter (7.1.4) temporarily attached to the inlet of the cassette. The mean value of these two measurements shall be used to calculate the total air volume sampled.

Basic strategies for monitoring environmental sources of airborne asbestos are described in annex G. After sampling, a cap shall be placed over the open end of the cassette, and the cassette packed with the filter face-upwards for return to the laboratory. Field blank filters shall also be included, as specified in 9.7, and submitted to the remaining analytical procedures along with the samples.

NOTES

5 In table 1 a collection filter area of 385 mm^2 is assumed, and the TEM grid openings are assumed to be 85 μm^2 square. The limit of detection is defined as the upper 95 % confidence limit of the Poisson distribution for a count of 0 structures. In the absence of background, this is equal to 2,99 times the analytical sensitivity. Backgrounds that are different from 0 observed during analysis of blank filters will degrade the limit of detection.

6 The analytical sensitivity S, expressed in number of structures per litre, is calculated using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

A_f is the active area, in square millimetres, of sample collection filter;

A_g is the mean area, in square millimetres, of grid openings examined;

k is the number of grid openings examined;

V is the volume of air sampled, in litres.

Table 1 — Examples of the minimum number of grid openings required to achieve a particular analytical sensitivity and limit of detection

Analytical sensitivity	Limit of detection	Volume of air sampled (litres)					
structures/l	structures/l	500	1 000	2 000	3 000	4 000	5 000
0,1	0,30	1 066	533	267	178	134	107
0,2	0,60	533	267	134	89	67	54
0,3	0,90	356	178	89	60	45	36
0,4	1,2	267	134	67	45	34	27
0,5	1,5	214	107	54	36	27	22
0,7	2,1	153	77	39	26	20	16
1,0	3,0	107	54	27	18	14	11
2,0	6,0	54	27	14	9	7	6
3,0	9,0	36	18	9	6	5	4
4,0	12	27	14	7	5	4	4
5,0	15	22	11	6	4	4	4
7,0	21	16	8	4	4	4	4
10	30	11	6	4	4	4	4

9 Procedure for analysis

9.1 General

The techniques used to prepare TEM specimens are different for polycarbonate and cellulose ester filters. The preparation method to be used shall be either 9.3 or 9.4, depending on the type of membrane filter used for air sampling. Cleaning of the sample cassettes before they are opened, preparation of the carbon evaporator, criteria for acceptable specimen grids, and the requirement for blank determinations are identical for the two preparation techniques. TEM examination, structure counting, fibre identification and reporting of results are independent of the type of filter or preparation technique used.

The ability to meet the blank sample criteria is dependent on the cleanliness of equipment and supplies. Consider all supplies such as microscope slides and glassware as potential sources of asbestos contamination. It is necessary to wash all glassware before it is used. Wash any tools or glassware which come into contact with the air sampling filters or TEM specimen preparations both before use and between handling of individual samples. Where possible, disposable supplies should be used.

9.2 Cleaning of sample cassettes

Asbestos fibres can adher to the exterior surfaces of air sampling cassettes, and these fibres can be inadvertently transferred to the sample during handling. To prevent this possibility of contamination, and after ensuring that the cassette is tightly sealed, wipe the exterior surfaces of each sampling cassette before it is placed in the clean facility or laminar flow hood.

9.3 Direct preparation of TEM specimens from polycarbonate filters

9.3.1 Selection of filter area for carbon coating

Use a cleaned microscope slide to support representative portions of polycarbonate filter during the carbon evaporation. Double-coated adhesive tape is used to attach the filter portions to the glass slide. Take care not to stretch the polycarbonate filters during handling. Using freshly cleaned tweezers, remove the polycarbonate filter from the sampling cassette, and place it on to a second cleaned glass microscope slide which is used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut the filter by rocking the blade from the point, pressing it into contact with the filter. Repeat the process as necessary. Several such portions may be mounted on the same microscope slide. The scalpel blade and tweezers shall be washed and dried between the handling of each filter. Identify the filter portions by writing on the glass slide.

9.3.2 Carbon coating of filter portions

Place the glass slide holding the filter portions on the rotation-tilting device, approximately 10 cm to 12 cm

from the evaporation source, and evacuate the evaporator chamber (7.3.5) to a vacuum better than 0,013 Pa. The evaporation of carbon shall be performed in very short bursts, separated by a few seconds to allow the electrodes to cool. If evaporation of carbon is too rapid, the strips of polycarbonate filter will begin to curl, and cross-linking of the surface will occur. This cross-linking procedures a layer of polymer which is relatively insoluble in organic solvents, and it will not be possible to prepare satisfactory TEM specimens. The thickness of carbon required is dependent on the size of particles on the filter, and approximately 30 nm to 50 nm has been found to be satisfactory. If the carbon film is too thin, large particles will break out of the film during the later stages of preparation, and there will be few complete and undamaged grid openings on the specimen. Too thick a carbon film will lead to a TEM image which is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film thickness should be the minimum possible, while retaining most of the grid openings of the TEM specimen intact.

9.3.3 Preparation of the Jaffe washer

Place several pieces of lens tissue, as shown in figure 2, on the stainless steel bridge (7.1.3) and fill the washer (see 7.3.7) with chloroform (6.2) or 1-methyl-2-pyrrolidone (6.3) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.3.4 Placing of specimens in the Jaffe washer

Using a curved scalpel blade, cut three 3 mm square pieces of carbon-coated polycarbonate filter form the carbon-coated filter portion. Select three squares to represent the centre and the periphery of the active surface of the filter. Place each square of filter, carbon side up, on a TEM specimen grid, and place the grid and filter on the saturated lens tissue in the Jaffe washer. Place the three specimen grids from one sample on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. Cover the Jaffe washer with the lid, and allow the washer to stand for at least 8 h.

NOTE 7 It has been found that some polycarbonate filters will not completely dissolve in the Jaffe washer, even after exposure to chloroform for as long as 3 d. This problem is more severe if the surface of the filter was overheated during the carbon evaporation. It has been found that the problem of residual undissolved filter polymer can be overcome in several ways:

a) condensation washing of the grids, using chloroform as the solvent, after the initial Jaffe washer treatment, can often remove much of the residual filter medium in a period of approximately 30 min. To carry out this procedure, transfer the piece of lens tissue supporting the specimen grids to the cold finger of the condensation washer (7.3.8), which has achieved stable operating conditions. Operate the washer for approximately 30 min after inserting the grids;

b) used in a Jaffe washer, 1-methyl-2-pyrrolidone has been found to be a more effective solvent than chloroform for polycarbonate filters. This solvent is more effective if the lens paper is not used and grids are placed directly on the stainless steel mesh of the Jaffe washer. A dissolution period of 2 h to 6 h has been found to be satisfactory. After dissolution is complete, remove the stainless steel mesh from the Jaffe washer and allow the grids to dry. 1-methyl-2-pyrrolidone evaporates very slowly. If it is required to dry the grids more rapidly, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash;

c) a mixture of 20 % 1,2-diaminoethane [ethylenediamine] and 80 % 1-methyl-2-pyrrolidone, used in a Jaffe washer, completely dissolves polycarbonate filters in 15 min, even if the surface of the filter has been overheated. To use this solvent, place the grids directly on the stainless steel mesh of the Jaffe washer, do not use the lens paper. After a period of 15 min, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash.

9.3.5 Rapid preparation of TEM specimens from PC filters

TEM specimens can be prepared rapidly from PC filters, if desired, by washing for approximately 1 h in a Jaffe washer, followed by washing for 30 min in a condensation washer using chloroform as the solvent. The alternative filter dissolution procedures described in note 7 may also be used.

9.4 Direct preparation of TEM specimens from cellulose ester filters

9.4.1 Selection of area of filter for preparation

Using clean tweezers, remove the filter from the filter cassette, and place it on a cleaned microscope slide. Using a clean, curved scalpel blade, cut out a portion of the filter.

9.4.2 Preparation of solution for collapsing cellulose ester filters

Mix 35 ml of dimethylformamide (6.4), and 15 ml of glacial acetic acid (6.5) with 50 ml of water (6.1). Store this mixture in a clean bottle, The mixture is stable and suitable for use for up to 3 months after preparation.

9.4.3 Filter collapsing procedure

Using a micropipette with a disposable tip (7.3.14), place 15 $\mu l/cm^2$ to 25 $\mu l/cm^2$ of the solution prepared in 9.4.2 on a cleaned microscope slide, and using the end of the pipette tip, spread the liquid over the area to be occupied by the filter portion. Place the filter portion, active surface upwards, on top of the solution, lowering the edge of the filter at an angle of about 20° so that air bubbles are not created. Remove any solution not absorbed by the filter by allowing a paper tissue to contact the liquid at the edge of the filter. More than one filter portion may be placed on one slide. Place the slide either on a thermostatically controlled slide warmer (7.3.9) at a temperature of 65 °C to 70 °C, or in an oven (7.3.9) at this temperature, for 10 min. The filter collapses slowly to about 15 % of its original thickness. The procedure leaves a thin, transparent polymer film, with particles and fibres embedded in the upper surface.

9.4.4 Plasma etching of the filter surface

The optimum conditions and time for plasma etching (see 7.3.4) have been determined experimentally from the recovery of fine chrysotile fibrils on 0,8 µm pore size MEC filters. The conditions required in a particular plasma asher shall be established using the procedure specified in annex A. Place the microscope slide holding the collapsed filter portions in the plasma asher, and etch for the time and under the conditions determined. Take care to ensure that the correct conditions are respected. After etching, admit air slowly to the chamber and remove the microscope slide.

Adjust the air admission valve of the plasma asher such that the time taken for the chamber to reach atmospheric pressure exceeds 2 min. Rapid air admission may disturb particulates on the surface of the etched filter.

9.4.5 Carbon coating

Coat the microscope slide holding the collapsed filter portions with carbon as specified in 9.3.2.

9.4.6 Preparation of the Jaffe washer

Place several pieces of lens tissue on the stainless steel bridge, and fill the washer with dimethylformamide (6.4) or acetone (6.6) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.4.7 Placing of specimens in the Jaffe washer

Place the specimens in the Jaffe washer as specified in 9.3.4. Specimens are normally cleared after approximately 4 h.

9.4.8 Rapid preparation of TEM specimens from cellulose ester filters

An alternative washing procedure may be used to prepare TEM specimens from cellulose ester filters more rapidly than can be achieved by the Jaffe washing procedure. After the specimens have been washed in a Jaffe washer for approximately 1 h, transfer the piece of lens tissue supporting the specimens to the cold finger of a condensation washer (7.3.8) operating with acetone as the solvent because dimethylformamide shall not be used in a condensation washer. Operate the condensation washer for approximately 30 min. This treatment removes all the remaining filter polymer.

9.5 Criteria for acceptable TEM specimen grids

Valid data cannot be obtained unless the TEM specimens meet specified quality criteria. Examine the TEM specimen grid in the electron microscope at a sufficiently low magnification (× 300 to × 1 000) for complete grid openings to be inspected. Reject the grid if

a) the TEM specimen has not been cleared of filter medium by the filter dissolution step. If the TEM specimen exhibits areas of undissolved filter medium, and if at least two of the three specimen grids are not cleared, either additional washing with solvent shall be carried out, or new specimens shall be prepared from the filter;

b) the sample is overloaded with particulate. If the specimen grid exhibits more than approximately 10 % obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. This filter cannot be alanysed satisfactorily using the direct preparation methods because the grid is too heavily loaded with debris to allow separate examination of individual particles by ED and EDXA, and obscuration of fibres by

other particulates may lead to underestimation of the asbestos structure count;

c) the particulate deposits on the specimen are not uniformly distributed from one grid opening to the next. If the particulate deposits on the specimen are obviously not uniform from one grid opening to the next, the specimen shall be designated as non-uniform. This condition is a function either of the air sampling conditions or of the fundamental nature of the airborne particulate. Satisfactory analysis of this filter may not be possible unless a large number of grid openings is examined;

d) the TEM grid is too heavily loaded with fibrous structures to make an accurate count. Accurate counts cannot be made if the grid has more than approximately 7 000 structures/mm^2; or

e) more than approximately 25 % of the grid openings have broken carbon film over the whole grid opening. Since the breakage of carbon film is usually more frequent in areas of heavy deposit, counting of the intact openings can lead to an underestimate of the asbestos structure count.

NOTE 8 If the specimens are rejected because unacceptable numbers of grid openings exhibit broken carbon replica, an additional carbon coating may be applied to the carbon coated filter, and new specimen grids prepared. The larger particles can often be supported by using a thicker carbon film. If this action does not produce acceptable specimen grids, this filter cannot be analysed using the direct preparation methods.

If one or more of the conditions described in b), c), d) or e) exists, it may not be possible to analyse the sample by this method.

9.6 Procedure for structure counting by TEM

9.6.1 General

The examination consists of a count of asbestos structures which are present on a specified number of grid openings. Fibres are classified into groups on the basis of morphological observations, ED patterns and EDXA spectra. The total number of structures to be counted depends on the statistical precision desired. In the absence of asbestos structures, the area of the TEM specimen grids which must be examined depends on the analytical sensitivity required. The precision of the structure count depends not only on the total number of structures counted, but also on their uniformity from one grid opening to the next. Additional structure counting will be necessary if greater precision is required.

In order that the estimate of the structure density on the sampling filter shall not be based on the small area represented by one specimen grid, grid openings shall be examined on two of the three specimen grids prepared. Then combine the results in the calculation of the structure density. Structure counts shall be made at a magnification of approximately × 20 000, and shall be terminated at the end of the examination of the grid opening on which the 100th asbestos structure is observed, except that the count shall be continued until a minimum of 4 grid openings have been examined. Otherwise, the structure count shall continue to that number of grid openings at which the specified analytical sensitivity has been achieved.

NOTE 9 The normal range for the number of grid openings which should be examined is from 4 to 20. If insufficient air has been sampled through the filter, the calculation in 9.6.4 can indicate that an impractically large number of grid openings should be examined. When this situation occurs, a larger value of analytical sensitivity may have to be accepted.

9.6.2 Measurement of mean grid opening area

The mean grid opening area shall be measured for the type of TEM specimen grids in use. The standard deviation of the mean of 10 openings selected from 10 grids should be less than 5 %. As an optional procedure, or if the 5 % standard deviation criterion cannot be demonstrated, the dimensions of each grid opening examined in the TEM shall be measured at a calibrated magnification.

9.6.3 TEM alignment and calibration procedures

Before structure counting is performed, align the TEM according to instrumental specifications. Calibrate the TEM and EDXA system according to the procedures specified in annex B.

9.6.4 Determination of stopping point

Before structure counting is begun, calculate the area of specimen to be examined in order to achieve the selected analytical sensitivity. Calculate the maximum number of grid openings to be examined using the following equation:

$$k = \frac{A_f}{A_g VS}$$

where

k is the number of grid openings to be examined, rounded upwards to the next highest integer;

A_f is the area, in square millimetres, of sample filter;

A_g is the area, in square millimetres, of TEM specimen grid opening;

V is the volume of air sampled, in litres;

S is the required analytical sensitivity, expressed in number of structures per litre.

9.6.5 General procedure for structure counting and size analysis

Use at least two specimen grids prepared from the filter in the structure count. Select at random several grid openings from each grid, and combine the data in the calculation of the results.

Use a form similar to that shown in figure 4 to record the data. Insert the first specimen grid into the TEM.

NOTE 10 In order to facilitate quality assurance measurements which require re-examination of the same grid opening by different microscopists, the grid should be inserted into the specimen holder in a standard orientation with the grid bars parallel and perpendicular to the axis of the specimen holder. This will provide scan directions parallel to the edges of the grid opening. It should be ensured that all microscopists begin scanning at the same starting point on the grid opening, and that they use similar scan patterns. This procedure permits rapid relocation of fibrous structures for further examination if necessary.

Select a typical grid opening and set the screen magnification to the calibrated value (approximately × 20 000). Adjust the sample height until the features in the centre of the TEM viewing screen are at the eucentric point. Set the goniometer tilt angle to zero. In column 1 of the data recording form, record the number or letter used to identify the grid. In column 2, record the identification of the particular grid opening. Position the specimen so that the grid opening is positioned with one corner visible on the screen. Move the image by adjustment of only one translation control, carefully examining the sample for fibres, until the opposite side of the grid opening is encountered. Move the image by a predetermined distance less than one screen diameter, using the other translation control, and scan the image in the reverse direction. Continue the procedure in this manner until the entire grid opening has been inspected in a pattern similar to that shown in figure 5. When a fibrous structure is detected, assign a sequential number to the primary structure in column 3, perform the identification procedures required as detailed in annex E, and enter the appropriate compositional classification on the structure counting form in column 5. Assign a morphological classification to the structure according to the procedures specified in annex D, and record this in column 6. Measure on the TEM viewing screen the length and width of the image of the primary structure, in millimetres, and record these measurements in columns 7 and 8. For a disperse cluster or matrix, assign a compositional classification and a morphological classification to each structure component, measure the length and width, and enter the data in columns 4 to 8. Use column 4 of the data recording form to tabulate the sequential number of total structures taking into account structure components. If non-asbestos fibres are observed, note their presence and type, if known. After a fibrous structure has been examined and measured, relocate the original field of view accurately before continuing scanning of the specimen. Failure to do this may cause structures to be overlooked or counted twice. Continue the examination until the completion of the grid opening on which the 100th asbestos structure has been recorded, or until the number of grid openings required to achieve the specified analytical sensitivity, calculated according to 9.6.4, have been examined whichever occurs first. The data shall be drawn approximately equally from a minimum of two grids. Regardless of the value calculated according to 9.6.4, fibrous structures on a minimum of four openings shall be counted.

9.6.6 Measurement of concentration for asbestos fibres and bundles longer than 5 µm

Consider improving the statistical validity for measurement of asbestos fibres and bundles longer than 5 µm by additional examination at a lower magnification, taking account only of the longer fibres and bundles. Perform this extended examination for fibres and bundles longer than 5 µm in accordance with the procedures specified in annex E. Use a magnification of approximately × 10 000 for counting all asbestos fibres and bundles longer than 5 µm, or approximately × 5 000 if only fibres and bundles within the diameter range 0,2 µm to 3,0 µm are to be counted. Continue the count until completion of the grid opening on which 100 fibres and bundles have been recorded, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity. Only those structures which are identified as, or are suspected to be, either chrysotile or one of the amphibole minerals will be reported in either the original or the extended TEM examination. Other materials, such as gypsum, cellulose fibres, and filter artifacts such as undissolved filter strands, will not be included in the fibre count. This restriction is intended to ensure that the best statistical validity is obtained for the materials of interest.

TEM asbestos structure count (page of)

Report number: .....
Sample number: .....
File name: .....
Sample description: .....
.....
.....
Preparation date: ..... By: .....
Analysis date: ..... By: .....
Computer entry date: ..... By: .....

Air volume: ..... litres
Sample filter area: ..... mm^2
Magnification: .....
Grid opening dimension: ..... µm
Level of analysis (C): .....
(A): .....

Grid	Grid opening	Number of structures		Class	Type of structure	Length	Width	Comments
		primary	total			mm	mm	

Figure 4 — Example of structure counting form

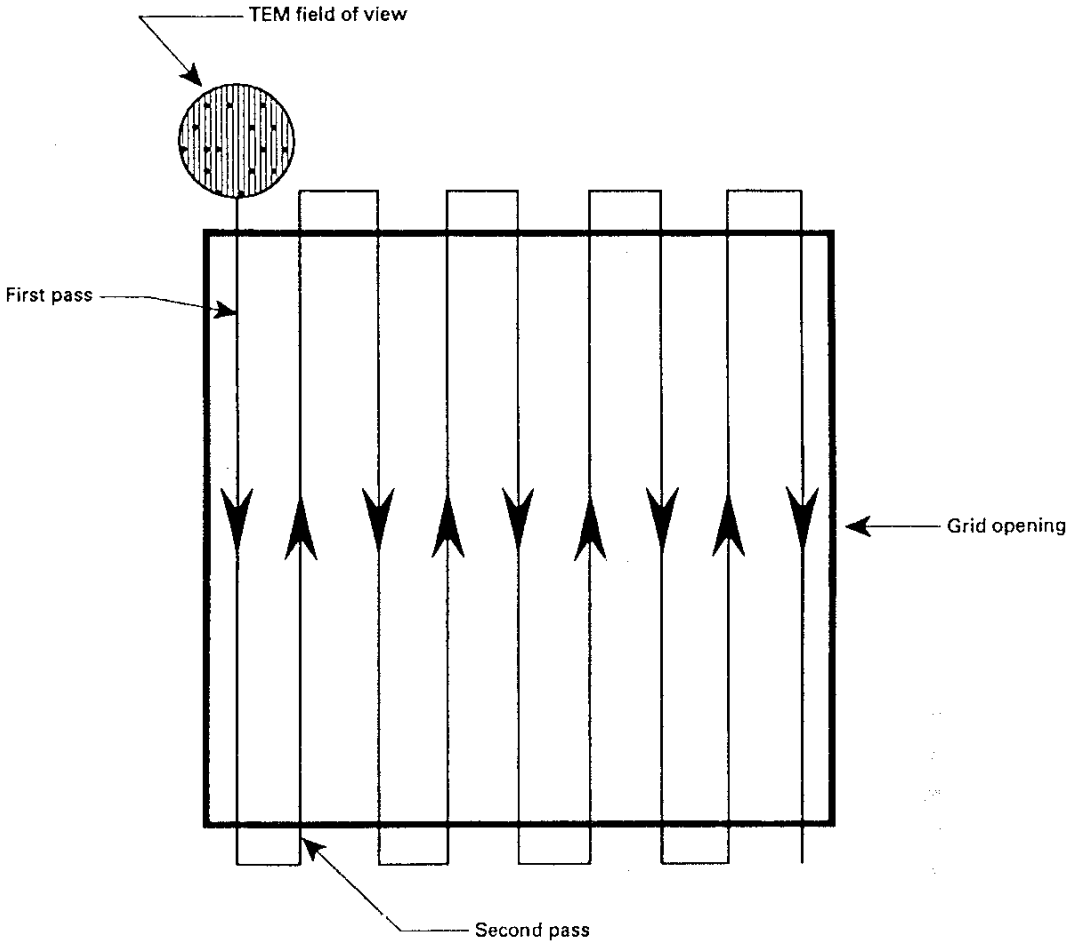


Figure 5 — Example of scanning procedure for TEM specimen examination

9.7 Blank and quality control determinations

Before air samples are collected, a minimum of two unused filters from each filter lot of 100 filters shall be analysed to determine the mean asbestos structure count. If the mean count for all types of asbestos structures is found to be more than 10 structures/mm^2, or if the mean fibre count for asbestos fibres and bundles longer than 5 µm is more than 0,1 fibre/mm^2, reject the filter lot.

To ensure that contamination by extraneous asbestos fibres during specimen preparation is insignificant compared with the results reported on samples, establish a continuous programme of blank measurements. At least one field blank shall be processed along with each batch of samples. In addition, at least one unused filter shall be included with every group of samples prepared on one microscope slide.

Initially, and also at intervals afterwards, ensure that samples of known asbestos concentrations can be analysed satisfactorily. Since there is a subjective component in the structure counting procedure, it is necessary that recounts of some specimens be made by different microscopists, in order to minimize the subjective effects. Such recounts provide a means of maintaining comparability between counts made by different microscopists. Variability between and within microscopists and between laboratories shall be characterized. These quality assurance measurements shall constitute approximately 10 % of the analyses. Repeat results should not differ at the 5 % significance level.

9.8 Calculation of results

Calculate the results using the procedures detailed in annex F. Prior to the TEM examination of the specimens, the level of analysis was specified. Before the results are calculated, the compositional and morphological classifications to be included in the result shall be specified. The chi-squared uniformity test shall be conducted using the number of primary asbestos structures found on each grid opening, prior to the application of the cluster and matrix counting criteria. The concentration result shall be calculated using the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

10 Performance characteristics

10.1 General

It is important to use this analytical method in conjunction with a continuous quality control programme. The quality control programme should include use of standard samples, blank samples, and both interlaboratory and intralaboratory analyses.

10.2 Interferences and limitations of fibre identification

Unequivocal identification of every chrysotile fibre is not possible, due to both instrumental limitations and the nature of some of the fibres. The requirement for a calibrated ED pattern eliminates the possibility of an incorrect identification of the fibre selected. However, there is a possibility of misidentification of fibres for which both the morphologies and the ED patterns are reported on the basis of visual inspection only. The only significant possibilities of misidentification occur with halloysite, vermiculite scrolls or palygorskite, all of which can be discriminated from chrysotile by the use of EDXA and by observation of the 0,73 nm (002) reflection of chrysotile in the ED pattern.

As in the case of chrysotile fibres, complete identification of every amphibole fibre is not possible due to instrumental limitations and the nature of some of the fibres. Moreover, complete identification of every amphibole fibre is not practical due to the limitations of both time and cost. Particles of a number of other minerals with compositions similar to those of some amphiboles could be erroneously classified as amphibole when the classification criteria do not include zone-axis ED techniques. However, the requirement for quantitative EDXA measurements on all fibres as support for the random orientation ED technique makes misidentification very unlikely, particularly when other similar fibres in the same sample have been identified as amphibole by zone-axis methods. The possibility of misidentification is further reduced with increasing aspect ratio, since it is rare for the minerals with which amphibole may be confused to display an asbestiform habit.

10.3 Precision and accuracy (see ISO Standard Handbook No. 3)

10.3.1 Precision

The analytical precision that can be obtained is dependent upon the number of structures counted, and also on the uniformity of the particulate deposit on the original filter. Assuming that the structures are randomly deposited on the filter, if 100 structures are counted and the loading is at least 3,5 structures/grid opening, computer modelling of the counting procedure shows that a coefficient of variation of about 10 % can be expected. As the number of structures counted decreases, the precision will also decrease approximately as $\sqrt{N}$, where N is the number of structures counted. In practice, particulate deposits obtained by filtration of ambient air samples are rarely ideally distributed, and it is found that the precision is correspondingly reduced. Degradation of precision is a consequence of several factors, such as:

a) non-uniformity of the filtered particulate deposit;

b) distorsion of the fibre distribution by application of the structure counting criteria;

c) variation between microscopists in their interpretation of the fibrous structures;

d) variation between microscopists in their ability to detect and identify fibres.

The 95 % confidence interval about the mean for a single structure concentration measurement using this analytical method should be approximately ± 25 % when 100 structures are counted over 10 grid openings.

10.3.2 Accuracy

There is no independent method available to determine the accuracy.

NOTE 11 It has been demonstrated that, after polycarbonate membrane filters have been coated with carbon, particulate material is transferred to the TEM specimens without measurable losses. However, if the filters are heavily loaded by particulate material, some of this may be lost before they are coated with carbon. Good comparability between the capillary-pore polycarbonate pro-

cedure and the cellulose ester filter procedure has been demonstrated for laboratory-generated aerosols of chrysotile asbestos.

10.3.3 Interlaboratory and intralaboratory analyses

Interlaboratory and intralaboratory analyses are required in order to monitor systematic errors that may develop among microscopists when using this method. These analyses should be designed to test both the overall method and the performance of individual microscopists. Repeating preparation of TEM grids from different sectors of a filter, followed by examination of the grids by a different microscopist, is a test for the reproducibility of the whole method. However, non-uniformity of the particulate deposit on the filter may lead to differences which are not related to the performance of the microscopists. Verified fibre counting (counting of asbestos structures on the same grid opening of a TEM grid by two or more operators, followed by resolution of any discrepancies) may be used both as a training aid and to determine the performance of different microscopists. The use of indexed TEM grids as described in 7.4.1 and 7.4.2 is recommended in order to facilitate relocation of specific grid openings.

10.4 Limit of detection

The limit of detection of the method can be varied by choice of the area of the collection filter, the volume of air sampled and the area of the specimen examined in the TEM. It is also a function of the background of asbestos structures on unused filters. A limit of detection shall be quoted for each sample analysis.

NOTE 12 In practice, the lowest limit of detection is frequently determined by the total suspended particulate concentration, since each particle on the filter must be separated from adjacent ones by a distance large enough for the particle to be identified without interference. Particulate loadings on sampling filters greater than 25 $\mu g/cm^2$ usually preclude preparation of TEM specimens by the direct methods. If the analysis is to be performed with an acceptable expenditure of time, the area of the specimen examined in the TEM for structures of all sizes is limited in most cases to between 10 and 20 grid openings. In typical ambient or building atmospheres, it has been found that an analytical sensitivity of 1 structure/l can be achieved. In some circumstances, where the atmosphere is exceptionally clean, this can be reduced to 0,1 structure/l or lower. For fibres and bundles longer than 5 µm, the reduced magnifications specified permit larger areas of the TEM specimens to be examined with an acceptable expenditure of time, resulting in proportionately lower limits of detection. If no structures are found in the analysis, the upper 95 % confidence limit can be quoted as the upper boundary of the concentration, corresponding to 2,99 times the analytical sensitivity if a Poisson distribution of structures on the filter is assumed. This 95 % confidence limit for 0 structures counted is taken as the detection limit. Since there is sometimes contamination of unused samples filters by asbestos structures, this should also be taken into account in the discussion of limits of detection.

11 Test report

The test report shall include at least the following information:

a) reference to this International Standard;

b) identification of the sample;

c) the date and time of sampling, and all necessary sampling data;

d) the date of the analysis;

e) the identity of the analyst;

f) any procedure used that is not specified in this International Standard or regarded as optional;

g) a complete listing of the structure counting data (the following data should be included: grid opening number, structure number, identification category, structure type, length and width of the structure in micrometres, and any comments concerning the structure);

h) a statement of the minimum acceptable identification category and the maximum identification category attempted (refer to tables D.1 and D.2);

i) a statement specifying which identification and structure categories have been used to calculate the concentration values;

j) separate concentration values for chrysotile and amphibole structures, expressed in number of asbestos structures per litre;

k) the 95 % confidence interval limits for the concentration values, expressed in number of asbestos structures per litre;

l) the analytical sensitivity, expressed in number of asbestos structures per litre;

m) the limit of detection, expressed in number of asbestos structures per litre;

n) compositional data for the principal varieties of amphibole, if present;

o) items g) to m) for asbestos fibres and bundles longer than 5 µm;

p) items g) to m) for PCM equivalent asbestos fibres and bundles.

An example of a suitable format for the structure counting data is shown in figures 6 and 7.

Sample analysis information (page 1)

Laboratory name		**Report number**	**Date**
Sample:	456 Queen Street Ashby de la Zouch Exterior sample 1991-09-09		
Air volume:			2 150,0 litres
Area of collection filter:			385,0 mm^2
Level of analysis (chrysotile):			CD or CMQ
Level of analysis (amphibole):			ADQ
Magnification used for fibre counting:			× 20 500
Aspect ratio for fibre definition:			5/1
Mean dimension of grid openings:			95,4 µm
Initials of analyst:			JMW
Number of grid openings examined:			10
Analytical sensitivity:			1,968 structures/l
Number of primary asbestos structures:			13
Number of asbestos structures counted:			26
Number of asbestos structures > 5 µm :			7
Number of asbestos fibres and bundles > 5 µm :			10
Number of PCM equivalent asbestos structures:			3
Number of PCM equivalent asbestos fibres:			5

Figure 6 — Example of format for reporting sample and preparation data

Sample analysis information (pages 2 and following)

Laboratoriy name **Report number** **Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

TEM asbestos structure count — Raw data

Grid	Grid opening	Number of structures		Identification[1]	Structure type	Length	Width	Comments
		primary	total			µm	µm	
A	F4-4	1	1	CD	F	1,7	0,045	
		2	2	CMQ	B	2,6	0,09	
		3	3	ADQ	F	4,0	0,15	Crocidolite
	E3-6	4	4	CD	MC+0	3,5	1,3	
	E5-1	5		CD	MD43	7,5	5,0	
			5	CD	MB	7,7	0,30	
			6	CMQ	MF	5,6	0,045	
			7	CD	MB	5,1	0,30	
			8	CD	MF	1,7	0,045	
B	F4-1	6		CD	CD+0	6,5	3,0	
			9	CD	CB	3,5	0,15	
			10	CD	CF	3,5	0,045	
			11	CMQ	CR+0	2,6	1,9	
	G5-1	7		CD	CD31	6,1	3,2	
			12	CD	CB	5,6	0,3	
			13	CMQ	CF	4,0	0,045	
			14	CMQ	CB	3,2	0,090	
	E4-4	8	15	CD	B	1,5	0,23	
		9	16	AD	F	8,7	0,15	
C	G4-4	10		CMQ	CD42	25	5,6	
			17	CMQ	CB	15	0,15	
			18	CMQ	CF	9,4	0,045	
			19	ADQ	CF	3,6	0,30	Tremolite
			20	CM	CF	4,2	0,045	
	E4-4			No fibres				
	E5-6	11		ADQ	CD+3	9,4	2,5	
			21	ADQ	CF	7,1	0,30	Amosite
			22	ADQ	CF	6,2	0,10	Crocidolite
			23	CM	CB	5,1	0,2	
			24	CM	CR+0	3,3	1,8	
	F4-1	12	25	CMQ	MC10	3,7	2,1	
		13	26	CD	CC+0	7,4	0,5	

1) Identification codes listed in tables D.1 and D.2.

Figure 7 — Example of format for reporting structure counting data

Annex A
(normative)

Determination of operating conditions for plasma asher

A.1 General

During the preparation of TEM specimens from an MEC or cellulose nitrate filter, the spongy structure of the filter is collapsed into a thinner film of polymer by the action of a solvent. Some of the particles on the surface of the original filter become completely buried in the polymer, and the specimen preparation procedure incorporates a plasma etching step to oxidize the surface layer of the polymer. Particles buried by the filter collapsing step are then exposed so that they can become subsequently affixed to the evaporated carbon film without altering their position on the original filter. The amount of etching is critical, and individual ashers vary in performance. Therefore, the plasma asher (7.3.4) shall be calibrated to give a known amount of etching of the surface of the collapsed filter. This is carried out by adjusting the radio-frequency power output and the oxygen flow-rate, and measuring the time taken to completely oxidize an uncollapsed cellulose ester filter with 25 mm diameter of the same type and pore size as those used in the analysis.

A.2 Procedure

Place an unused cellulose ester filter, with 25 mm diameter, of the same type as that being used, in the centre of a glass microscope slide. Position the slide approximately in the centre of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 ml/min to 20 ml/min. Adjust the tuning of the system so that the intensity of the plasma in maximized. Measure the time required for complete oxidation of the filter. Determine operating parameters which result in complete oxydation of the filter in a period of approximately 15 min. For etching of collapsed filters, these operating parameters shall be used for a period of 8 min.

NOTE 13 Plasma oxidation at high radio-frequency powers will cause the filter to shrink and curl, followed by sudden violent ignition. At lower powers, the filter will remain in position and will slowly become thinner until it is nearly transparent. It is recommended that a radio-frequency power be used such that violent ignition does not occur. When multiple filters are etched, the rate of etching is reduced, and the system should be calibrated accordingly.

Annex B
(normative)

Calibration procedures

B.1 Calibration of the TEM

B.1.1 Calibration of TEM screen magnification

The electron microscope should be aligned according to the specifications of the manufacturer. Initially, and at regular intervals, calibrate the magnifications used for the analysis using a diffraction grating replica (7.3.11). Adjust the specimen height to the eucentric position before carrying out the calibration. Measure the distance on the fluorescent viewing screen occupied by a convenient number of repeat distances of the grating image, and calculate the magnification. Always repeat the calibration after any instrumental maintenance or change of operating conditions. The magnification of the image on the viewing screen is not the same as that obtained on photographic plates or film. The ratio between these is a constant value for the particular model of TEM.

B.1.2 Calibration of ED camera constant

Calibrate the camera constant of the TEM when used in ED mode. Use a specimen grid supporting a carbon film on which a thin film of gold has been evaporated or sputtered. Form an image of the gold film with the specimen adjusted to the eucentric position and select ED conditions. Adjust the objective lens current to optimize the pattern obtained, and measure the diameters of the innermost two rings either on the fluorescent viewing screen or on a recorded image. Calculate the radius-based camera constant, λL, for both the fluorescent screen and the photographic plate or film, using the following equation:

$$\lambda L = \frac{aD}{2{,}0\sqrt{h^2 + k^2 + l^2}}$$

where

λ is the wavelength, in nanometres, of the incident electrons;

L is the camera length, in millimetres;

a is the unit cell dimension of gold, in nanometres (= 0,407 86 nm);

D is the diameter, in millimetres, of the (hkl) diffraction ring.

Using gold as the calibration material, the radius-based camera constant is given by

$\lambda L = 0{,}117\ 74D$ mm·nm (smallest ring)

$\lambda L = 0{,}101\ 97D$ mm·nm (second ring)

B.2 Calibration of the EDXA system

Energy calibration of the EDXA system for a low energy and high energy peak shall be performed regularly. Calibration of the intensity scale of the EDXA system permits quantitative composition data, at an accuracy of about 10 % of the elemental concentration, to be obtained from EDXA spectra of reference silicate minerals involving the elements Na, Mg, Al, Si, K, Ca, Mn and Fe, and applicable certified reference materials. If quantitative determinations are required for minerals containing other elements, reference standards other than those referred to below will be required. Well-characterized mineral standards permit calibration of any TEM-EDXA combination which meets the instrumental specifications of 7.3.1 and 7.3.2, so that EDXA data from different instruments can be compared. Reference minerals are required for the calibration; the criteria for selection being that they should be silicate minerals with matrices as close as possible to those of the amphiboles or serpentine, and that small individual fragments of the minerals are homogeneous in composition within a few percent.

Determine the compositions of these standards by electron microprobe analysis or chemicals methods. Crush fragments of the same selected mineral standards and prepare filters by dispersal of the crushed material in water and immediate filtration of the suspensions. Prepare TEM specimens from these filters according to the procedures specified in clause 9. These TEM specimens can then be used to calibrate any TEM-EDXA system so that comparable composi-

tional results can be obtained from different instruments.

NOTES

14 The microprobe analysis of the mineral standards are carried out by conventional techniques which can be found in annex J. The mineral is first embedded in a mount of poly(methyl methacrylate) or epoxy resin. The mount is then ground and polished to achieve a flat, polished surface of the mineral fragment. This surface is then analysed, using suitable reference standards, preferably oxide standards of the individual elements wherever these are available. It is necessary to take into account the water concentration in the minerals, which in the case of chrysotile amounts to 13 % by mass. This water content may vary due to losses in the vacuum system.

15 Aqueous suspensions of mineral standards should be filtered immediately after preparation, since alkali and alkali earth metals may be partially leached from minerals containing these elements.

Express the results of the electron microprobe analyses as atomic or mass percentage ratios relative to silicon. X-ray peak ratios of the same elements relative to silicon, obtained from the EDXA system, can then be used to calculate the relationship between peak area ratio and atomic or mass percentage ratio. The technique was described by Cliff and Lorimer (see annex J, reference [8]).

The X-rays generated in a thin specimen by an incident electron beam have a low probability of interacting with the specimen. Thus, mass absorption and fluorescence effects are negligible. In a silicate mineral specimen containing element i, the following equation can be used to perform quantitative analyses in the TEM:

$$\frac{C_i}{C_{Si}} = k_i \times \frac{A_i}{A_{Si}}$$

where

C_i is the concentration or atomic percentage of element i;

C_{Si} is the concentration or atomic percentage of silicon;

A_i is the elemental integrated peak area for element i;

A_{Si} is the elemental integrated peak area for silicon;

k_i is the k-ratio for element i relative to silicon.

For a particular instrumental configuration and a particular particle size, the value of k_i is constant.

To incorporate correction for the particle size effect on peak area ratios (see annex J, references [35] and [36], extend the Cliff and Lorimer technique by obtaining separate values of the constant k_i for different ranges of fibre diameter. It is recommended that 20 EDXA measurements be made for each range of fibre diameters. Suitable ranges of fibre diameter are:

$< 0{,}25$ µm; 0,25 µm to 0,5 µm; 0,5 µm to 1,0 µm; $> 1{,}0$ µm.

Insert the TEM grid into the transmission electron microscope, obtain an image at the calibrated higher magnification of about × 20 000, and adjust the specimen height to the eucentric point. If the X-ray detector is a side-entry variety, tilt the specimen towards the X-ray detector. Select an isolated fibre or particle less than 0,5 µm in width, and accumulate an EDXA spectrum using an electron probe of suitable diameter. When a well-defined spectrum has been obtained, perform a background subtraction and calculate the background-corrected peak areas for each element listed, using energy windows centred on the peaks. Calculate the ratio of the peak area for each specified element relative to the peak area for silicon. All background-subtracted peak areas used for calibration shall exceed 400 counts.

Repeat this procedure for 20 particles of each mineral standard. Reject analyses of any obviously foreign particles. Calculate the arithmetic mean concentration to peak area ratio, k_i (k-ratio), for each specified element of each mineral standard and for each of the fibre diameter ranges. Periodic routine checks shall be carried out to ensure that there has been no degradation of the detector performance. These k-ratios are used to calculate the elemental concentrations of unknown fibres, using the Cliff and Lorimer relationship.

Annex C
(normative)

Structure counting criteria

C.1 General

In addition to isolated fibres, other assemblages of particles and fibres frequently occur in air samples. Groupings of asbestos fibres and particles, referred to as "asbestos structures", are defined as fibre bundles, clusters and matrices. The numerical result of a TEM examination depends largely on whether the analyst assigns such an assemblage of fibres as a single entity, or as the estimated number of individual fibres which form the assemblage. It is therefore important that a logical system of counting criteria be defined, so that the interpretation of these complex structures is the same for all analysts, and so that the numerical result is meaningful. Imposition of specific structure-counting criteria generally requires that some interpretation, partially based on uncertain information on health effects, be made of each asbestos structure found. It is not the intention of this International Standard to make any interpretations based on health effects, and it is intended that a clear separation shall be made between recording of structure counting data, and later interpretation of those data. The system of coding specified in this International Standard permits a clear morphological description of the structures to be recorded in a concise manner suitable for later interpretation, if necessary, by a range of different criteria, without the necessity for re-examination of the specimens. In particular, the coding system is designed to permit the dimensions of each complex fibrous structure, and also whether these structures contain fibres longer than 5 µm, to be recorded. This approach permits later evaluations of the data to include considerations of particle respirability and comparisons with historical indices of asbestos exposure. Examples of the various types of morphological structure, and the manner in which these shall be recorded, are shown in figure C.1.

C.2 Structure definitions and treatment

Each fibrous structure that is a separate entity shall be designated as a primary structure. Each primary structure shall be designated as a fibre, bundle, cluster or matrix.

C.2.1 Fibre

Any particle with parallel or stepped sides, of minimum length 0,5 µm, and with an aspect ratio of 5/1 or greater, shall be defined as a fibre. For chrysotile asbestos, the single fibril shall be defined as a fibre. A fibre with stepped sides shall be assigned a width equal to the average of the minimum and maximum widths. This average shall be used as the width in determination of the aspect ratio.

C.2.2 Bundle

A grouping composed of apparently attached parallel fibres shall be defined as a bundle, with a width equal to an estimate of the mean bundle width, and a length equal to the maximum length of the structure. The overall aspect ratio of the bundle may have any value, provided that it contains individual constituent fibres with aspect ratios equal to or greater than 5/1. Bundles may exhibit diverging fibres at one or both ends.

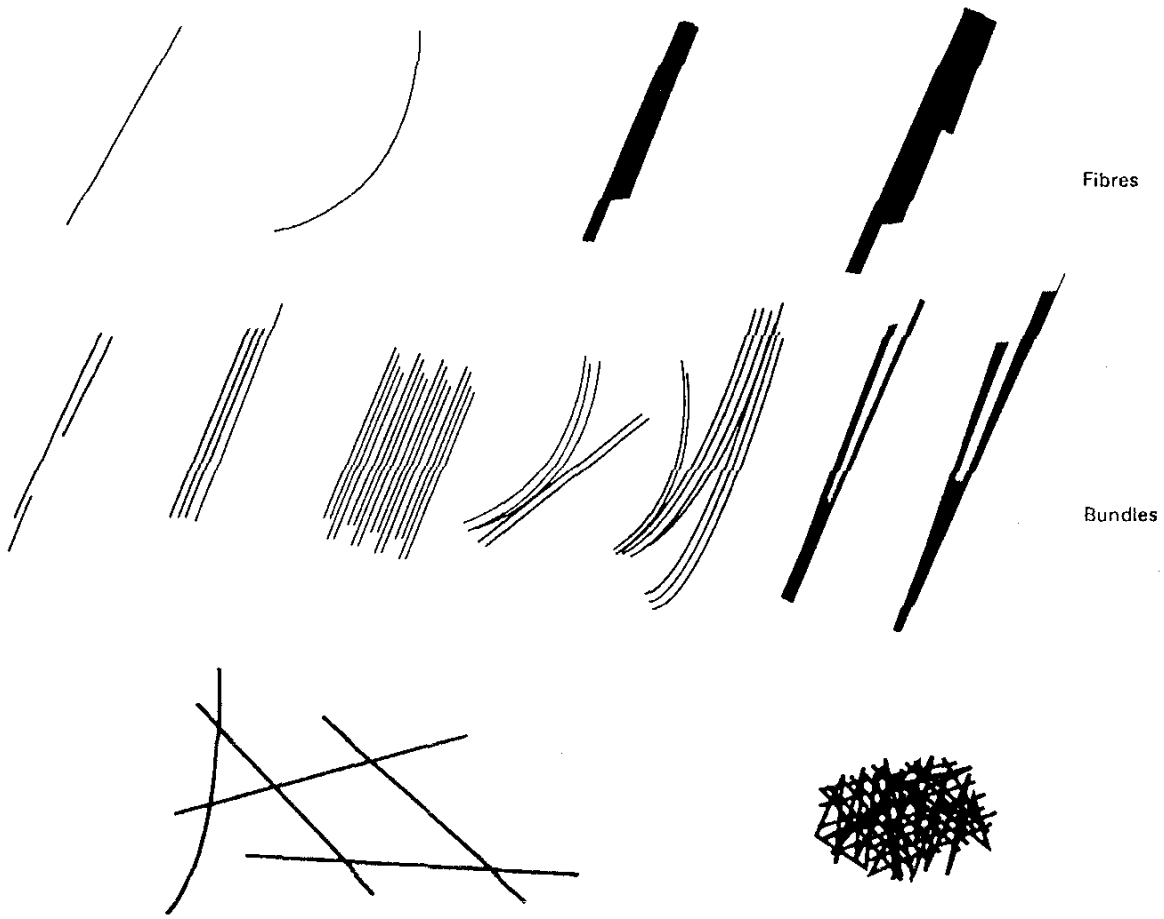
C.2.3 Cluster

An aggregate of two or more randomly oriented fibres, with or without bundles, shall be defined as a cluster. Clusters occur as two varieties.

C.2.3.1 disperse cluster (type D): Disperse and open network, in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured;

C.2.3.2 compact cluster (type C): Complex and tightly bound network, in which one or both ends of each individual fibre or bundle is (are) obscured, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, clusters can occur in which the characteristics of both types of cluster occur in the same structure. Where this occurs, the structure should be defined as a disperse cluster, and then a logical procedure should be followed by recording structure components according to the counting criteria. The procedure for treatment of clusters is illustrated by examples in figure C.2.



a) Disperse cluster (type D)

b) Compact cluster (type C)

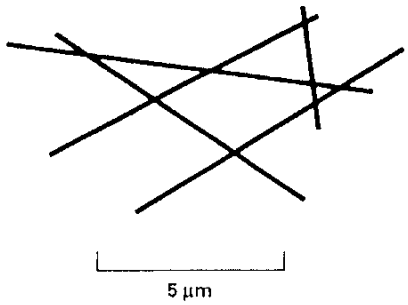
c) Disperse matrix (type D)

d) Compact matrix (type C)

Figure C.1 — Fundamental morphological structure types

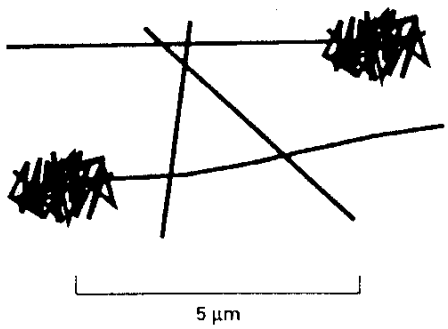
Count as 1 compact cluster containing more than 9 fibres (all fibres shorter than 5 µm)

Record as CC+0



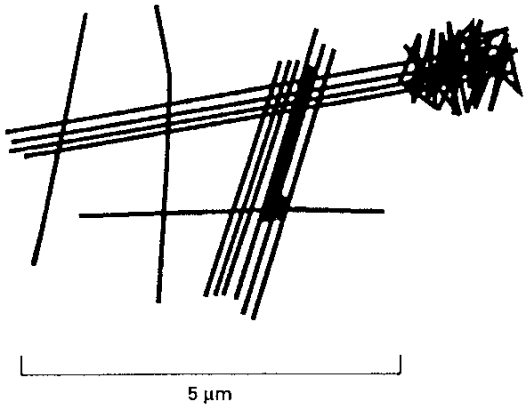
Count as 1 disperse cluster consisting of 5 fibres, 4 of which are longer than 5 µm

Record as CD54, followed by 5 fibres, each recorded as CF



Count as 1 disperse cluster consisting of 4 fibres, 2 of which are longer than 5 µm, and 2 cluster residuals, each containing more than 9 fibres

Record as CD+2, followed by 4 fibres, each recorded as CF, and 2 cluster residual, each recorded as CR+0



Count as 1 disperse cluster consisting of 3 fibres, 2 bundles, 1 of which is longer than 5 µm, and 1 cluster residual containing more than 9 fibres

Record as CD+1, followed by 3 fibres, each recorded as CF, 2 bundles, each recorded as CB, and 1 cluster residual recorded as CR+0

Figure C.2 — Examples of recording of complex asbestos clusters

C.2.4 Matrix

One or more fibres, or fibre bundles, may be attached to, or partially concealed by, a single particle or group of overlapping nonfibrous particles. This structure shall be defined as a matrix. The TEM image does not discriminate between particles which are attached to fibres, and those which have by chance overlapped in the TEM image. It is not known, therefore, whether such a structure is actually a complex particle, or whether it has arisen by a simple overlapping of particles and fibres on the filter.

Since a matrix structure may involve more than one fibre, it is important to define in detail how matrices shall be counted. Matrices exhibit different characteristics, and two types can be defined.

C.2.4.1 disperse matrix (type D): Structure consisting of a particle or linked group of particles, with overlapping or attached fibres or bundles in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured.

C.2.4.2 compact matrix (type C): Structure consisting of a particle or linked group of particles, in which fibres or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, matrices can occur in which the characteristics of both types of matrix occur in the same structure. Where this occurs, the structure should be assigned as a disperse matrix, and then a logical procedure should be followed by recording structure components according to the counting criteria. Examples of the procedure which shall be followed are shown in figure C.3.

C.2.5 Asbestos structure larger than 5 µm

Any fibre, bundle, cluster or matrix for which the largest dimension exceeds 5 µm. Asbestos structures larger than 5 µm do not necessarily contain asbestos fibres or bundles longer than 5 µm.

C.2.6 Asbestos fibre or bundle longer than 5 µm

An asbestos fibre of any width, or bundle of such fibres, which has a length exceeding 5 µm.

C.2.7 PCM equivalent structure

Any fibre, bundle, cluster or matrix with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. PCM equivalent structures do not necessarily contain fibres or bundles longer than 5 µm, or PCM equivalent fibres.

C.2.8 PCM equivalent fibre

Any particle with parallel or stepped sides, with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. For chrysotile, PCM equivalent fibres will always be bundles.

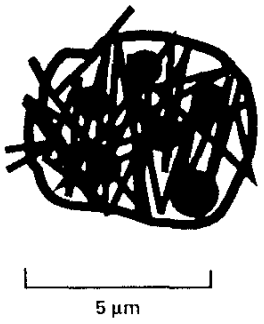
C.3 Other structure counting criteria

C.3.1 Structures which intersect grid bars

A structure which intersects a grid bar shall only be counted on two sides of the grid opening, as illustrated in figure C.4. Record the dimensions of the structure such that the obscured portions of components are taken to be equivalent to the unobscured portions, as shown by the broken lines in figure C.4. For example, the length of a fibre intersecting a grid bar is taken to be twice the unobscured length. Structures intersecting either of the other two sides shall not be included in the count.

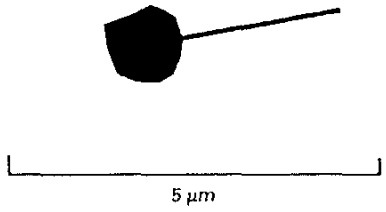
C.3.2 Fibres which extend outside the field of view

During scanning of a grid opening, count fibres which extend outside the field of view systematically, so as to avoid double-counting. In general, a rule should be established so that fibres extending outside the field of view in only two quadrants are counted. The procedure is illustrated by figure C.5. Measure the length of each of these fibre by moving the specimen to locate the other end of the fibre, and then return to the original field of view before continuing to scan the specimen. Fibres without terminations within the field of view shall not be counted.



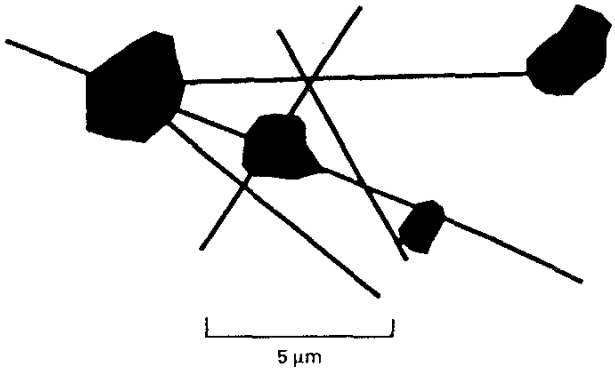
Count as 1 compact matrix, with all fibres shorter than 5 µm

Record as MC+0



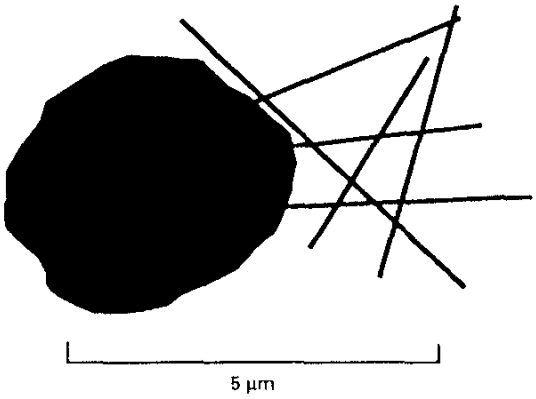
Count as 1 disperse matrix consisting of 1 fibre shorter than 5 µm

Record as MD10, followed by 1 fibre recorded as MF



Count as 1 disperse matrix consisting of 5 fibres, all longer than 5 µm

Record as MD55, followed by 5 fibres, each recorded as MF



Count as 1 disperse matrix, consisting if 3 fibres, 1 of which is longer than 5 µm, and 1 matrix residual containing 3 fibres

Record as MD61, followed by 3 fibres, each recorded as MF, and 1 matrix residual recorded as MR30

Figure C.3 — Examples of recording of complex asbestos matrices

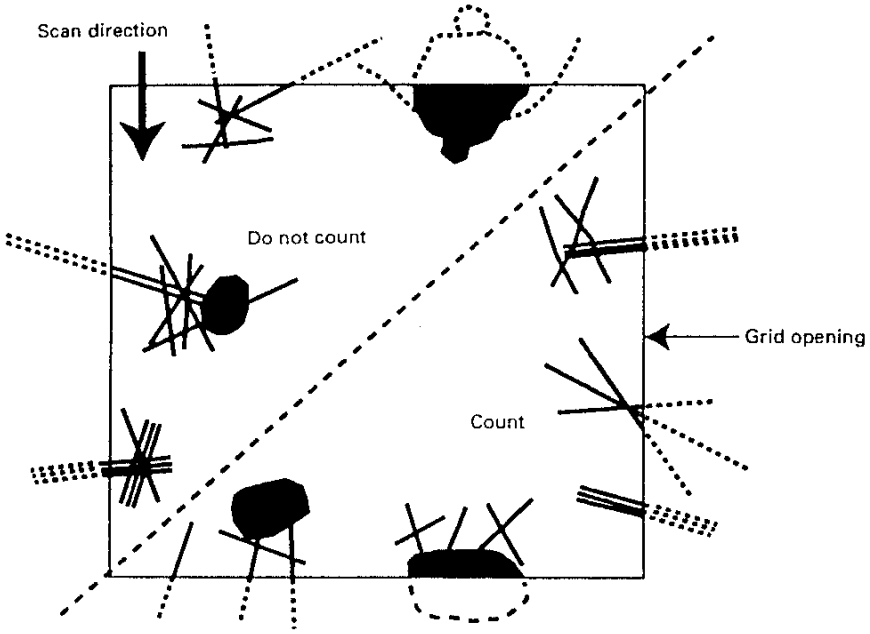


Figure C.4 — Example of counting of structures which intersect grid bars

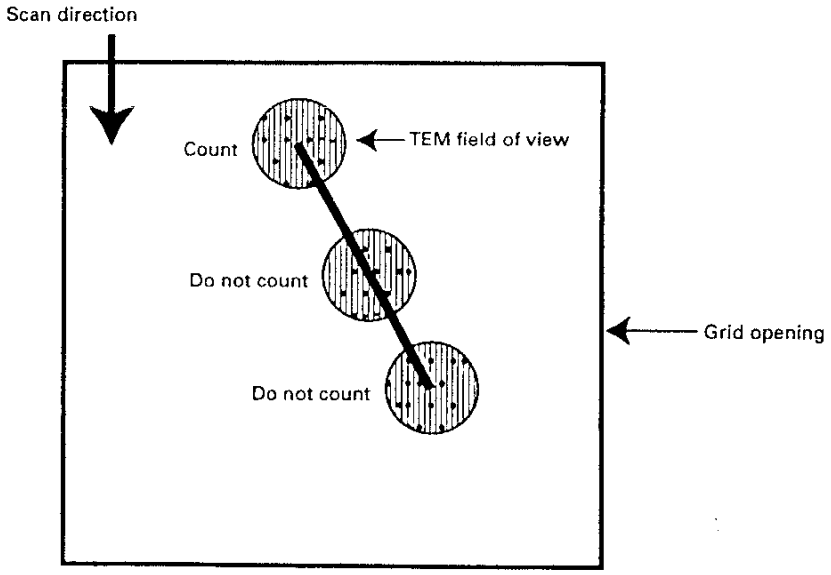


Figure C.5 — Example of counting of fibres which extend outside the field of view

C.4 Procedure for data recording

C.4.1 General

The morphological codes specified are designed to facilitate computer data processing, and to allow recording of a complete representation of the important features of each asbestos structure. The procedure requires that the microscopist classify each primary fibrous structure into one of the four fundamental categories: fibres, bundles, clusters and matrices.

C.4.2 Fibres

On the structure counting form, a fibre as defined in C.2.1 shall be recorded by the designation "F". If the fibre is a separately-counted part of a cluster or matrix, the fibre shall be recorded by the designation "CF", or "MF", depending on whether it is a component of a cluster or matrix.

C.4.3 Bundles

On the structure counting form, a bundle as defined in C.2.2 shall be recorded by the designation "B". If the bundle is a separately-counted part of a cluster or matrix, the bundle shall be recorded by the designation "CB", or "MB", depending on whether it is a component of a cluster or matrix.

C.4.4 Disperse clusters (type D)

On the structure counting form, an isolated cluster of type D as defined in C.2.3 shall be recorded by the designation "CD", followed by a two-digit number. The first digit represents the analyst's estimate of the total number of fibres and bundles comprising the structure. The digit shall be from 1 to 9, or designated as "+" if there are estimated to be more than 9 component fibres or bundles. The second digit shall represent, in the same manner, the total number of fibres and bundles longer than 5 µm contained in the structure. The overall dimensions of the cluster, in two perpendicular directions representing the maximum dimensions, shall be recorded. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "CF" (cluster fibre) and "CB" (cluster bundle). If, after accounting for prominent component fibres and bundles, a group of clustered fibres remains, this shall be recorded by the designation "CR" (cluster residual). If the remaining clustered fibres are present as more than one localized group, it may be necessary to record more than one cluster residual. Do not record more than 5 cluster residuals for any cluster. A cluster residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall cluster. Optionally, if the number of component fibres and bundles in either the original cluster or the cluster residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.5 Compact clusters (type C)

On the structure counting form, an isolated cluster of type C as defined in C.2.3 shall be recorded by the designation "CC", followed by a two-digit number. The two-digit number describing the numbers of component fibres and bundles shall be assigned in the same manner as for clusters of type D. The overall dimensions of the cluster in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact clusters cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.6 Disperse matrices (type D)

On the structure counting form, an isolated matrix of type D as defined in C.2.4 shall be recorded by the designation "MD", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "MF" (matrix fibre) and "MB" (matrix bundle). If after accounting for prominent component fibres and bundles, matrix material containing asbestos fibres remains, this shall be recorded by the designation "MR" (matrix residual). If the remaining matrix fibres are present as more than one localized group, it may be necessary to record more than one matrix residual. Do not record more than 5 matrix residuals for any matrix. A matrix residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall matrix. Optionally, if the number of component fibres or bundles in either the original matrix or the matrix residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.7 Compact matrices (type C)

On the structure counting form, an isolated matrix of type C as defined in C.2.4 shall be recorded by the

designation "MC", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact matrices cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.8 Procedure for recording of partially obscured fibres and bundles

The proportion of the length of a fibre or bundle that is obscured by other particulates shall be used as the basis for determining whether a fibre or bundle is to be recorded as a separate component or is to be considered as a part of a matrix of type C or part of a matrix residual. If the obscured length could not possibly be more than one-third of the total length, the fibre or bundle shall be considered a prominent feature to be separately recorded. The assigned length for each such partially obscured fibre or bundle shall be equal to the visible length plus the maximum possible contribution from the obscured portion. Fibres or bundles which appear to cross the matrix, and for which both ends can be located approximately, shall be included in the maximum of 5 and recorded according to the counting criteria as separate fibres or bundles. If the obscured length could be more than one third of the total length, the fibre or bundle shall be considered as a part of a compact matrix of type C or part of a matrix residual.

C.5 Special considerations for counting of PCM equivalent structures

Use 3/1 as the minimum aspect ratio for counting of PCM equivalent structures. This aspect ratio definition is required in order to achieve comparability of the results for this size range of structure with historical optical measurements, but use of this aspect ratio definition does not significantly affect the ability to interpret the whole fibre size distribution in terms of a minimum 5/1 aspect ratio. Some applications may require that a count be made of PCM equivalent structures only. The coding system permits discrimination between PCM equivalent structures that contain fibres and bundles longer than 5 µm and those that do not.

NOTE 16 In general, clusters and matrices will yield fewer components as the minimum dimensions specified for countable fibres are increased. Thus, it may be found that a particular structure yields a higher number of component fibres and bundles in a count for all fibre sizes than it does at a reduced magnification when only fibres and bundles longer than 5 µm are being counted. However, the requirement that component fibres and bundles be recorded in decreasing length order ensures that the data are consistent for a particular structure, regardless of the size category of fibres being counted and the magnification in use.

Annex D
(normative)

Fibre identification procedure

D.1 General

The criteria used for identification of asbestos fibres may be selected depending on the intended use of the measurements. In some circumstances, there can be a requirement that fibres shall be unequivocally identified as a specific mineral species. In other circumstances, there can be sufficient knowledge about the sample, so that rigorous identification of each fibre need not be carried out. The time required to perform the analysis, and therefore the cost of analysis, can vary widely depending on the identification criteria considered which are to be sufficiently definitive. The combination of criteria considered definitive for identification of fibres in a particular analysis shall be specified before the analysis is made, and this combination of criteria shall be referred to as the "level" of analysis. Various factors related to instrumental limitations and the character of the sample may prevent satisfaction of all of the specified fibre identification criteria for a particular fibre. Therefore, a record shall be made of the identification criteria which were satisfied for each suspected asbestos fibre included in the analysis. For example, if both ED and EDXA were specified to be attempted for definitive identification of each fibre, fibres with chrysotile morphology which, for some reason, do not give an ED pattern but which do yield an EDXA spectrum corresponding to chrysotile, are categorized in a way which conveys the level of confidence to be placed in the identification.

D.2 ED and EDXA techniques

D.2.1 General

Initially, fibres are classified into two categories on the basis of morphology: those fibres with tubular morphology, and those fibres without tubular morphology. Further analysis of each fibre is conducted using ED and EDXA methods. The following procedures should be used when fibres are examined by ED and EDXA.

The crystal structures of some mineral fibres, such as chrysotile, are easily damaged by the high current densities required for EDXA examination. Therefore, investigation of these sensitive fibres by ED should be completed before attempts are made to obtain EDXA spectra from the fibres. When more stable fibres, such as the amphiboles, are examined, EDXA and ED may be used in either order.

D.2.2 ED techniques

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel with the incident electron beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a "zone-axis ED pattern". In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but is is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone-axis. The angle between the two zone-axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when

the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or to be recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focussed in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it will be necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, an internal calibration standard shall be used. A thin coating of gold, or another suitable calibration material, shall be applied to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

If a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections the possibility of twinning or multiple diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in the references by J.A. Gard [11] P.B. Hirsch *et al* [14] and H.R. Wenck [42] included in annex J. Not all zone-axis patterns which can be obtained are definitive. Only those which have closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d-spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the first gold diffraction ring (111), and that patterns with smaller distances between reflections are usually the most definitive.

Five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern shall be selected for measurement, as shown in figure D.1. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very overexposed, it does not provide a well-defined origin for these measurements. The required distances shall therefore be obtained by measuring between pairs of spots symmetrically disposed about the centre spot preferably separated by several repeat distances. The distances shall be measured with a precision of better than 0,3 mm, and the angles to a precision of better than 2,5°. The diameter of the first or second ring of the calibration pattern (111 and 200) shall also be measured with a precision of better than 0,3 mm.

Using gold as the calibration material, the radius-based camera constant is given by

$$\lambda L = 0{,}117\ 74D \text{ mm·nm (first ring)}$$

$$\lambda L = 0{,}101\ 97D \text{ mm·nm (second ring)}$$

D.2.3 EDXA measurements

Interpretation of the EDXA spectrum may be either qualitative or quantitative. For qualitative interpretation of a spectrum, the X-ray peaks originating from the elements in the fibre are recorded. For quantitative interpretation, the net peak areas, after background subtraction, are obtained for the X-ray peaks originating from the elements in the fibre. This method provides quantitative interpretation for those minerals which contain silicon.

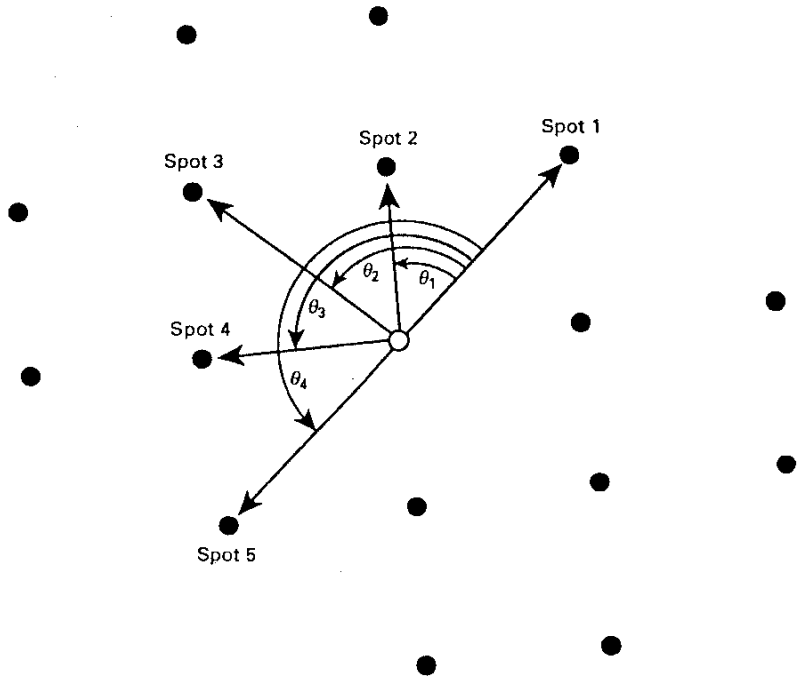


Figure D.1 — Example of measurement of zone-axis SAED patterns

To obtain an EDXA spectrum, move the image of the fibre to the centre of the screen and remove the objective aperture. Select an appropriate electron beam diameter and deflect the beam so that it impinges on the fibre. Depending on the instrumentation, it may be necessary to tilt the specimen towards the X-ray detector and, in some instruments, to use the Scanning Transmission Electron Microscopy (STEM) mode of operation.

The time for acquisition of a suitable spectrum varies with the fibre diameter, and also with instrumental factors. For quantitative interpretation, spectra should have a statistically valid number of counts in each peak. Analyses of small diameter fibres which contain sodium are the most critical, since it is in the low energy range that the X-ray detector is least sensitive. Consequently, it is necessary to acquire a spectrum for a period that is sufficiently long for the sodium to be detected in such fibres. It has been found that satisfactory quantitative an analyses can be obtained if acquisition is continued until the background subtracted silicon $K\alpha$ peak integral exceeds 10 000 counts. The spectrum should then be manipulated to subtract the background and to obtain the net areas of the elemental peaks.

After quantitative EDXA classification of some fibres by computer analysis of the net peak areas, it may be possible to classify further fibres in the same sample on the basis of comparison of spectra at the intrument. Frequently, visual comparisons can be made after somewhat shorter acquisition times.

D.3 Interpretation of fibre analysis data

D.3.1 Chrysotile

The morphological structure of chrysotile is characteristic, and with experience, can be recognized readily. However, a few holder minerals have a similar appearance, and morphological observation by itself is inadequate for most samples. The ED pattern obtained from chrysotile is quite specific for this mineral if the specified characteristics of the pattern correspond to those from reference chrysotile. However, depending on the past history of the fibre, and on a number of other factors, the crystal structure of a particular fibre may be damaged, and it may not yield and ED pattern. In this case, the EDXA spectrum may be the only data available to supplement the morphological observations.

D.3.2 Amphiboles

Since the fibre identification procedure for asbestos fibres other than chrysotile can be involved and time-consuming, computer programmes, such as that developed by B.L. Rhoades (see annex J, reference

[32]), are recommended for interpretation of zone-axis ED patterns. The published literature contains composition and crystallographic data for all of the fibrous minerals likely to be encountered in TEM analysis of air samples, and the compositional and structural data from the unknown fibre should be compared with the published data. Demonstration that the measurements are consistent with the data for a particular test mineral does not uniquely identify the unknown, since the possibility exists that data from other minerals may also be consistent. It is, however, unlikely that a mineral of another structural class could yield data consistent with that from an amphibole fibre identified by quantitative EDXA and two zone-axis ED patterns.

Suspected amphibole fibres should be classified initially on the basis of chemical composition. Either qualitative or quantitative EDXA information may be used as the basis for this classification. From the published data on mineral compositions, a list of minerals which are consistent in composition with that measured for the unknown fibre should be compiled. To proceed further, it is necessary to obtain the first zone-axis ED pattern, according to D.2.2.

It is possible to specify a particular zone-axis pattern for identification of amphibole, since a few patterns are often considered to be characteristic. Unfortunately, for a fibre with random orientation on a TEM grid, no specimen holder and goniometer currently available will permit convenient and rapid location of two preselected zone-axes. The most practical approach has been adopted, which is to accept those low index patterns which are easily obtained, and then to test their consistency with the structures of the minerals already preselected on the basis of the EDXA data. Even the structures of non-amphibole minerals in this preselected list shall be tested against the zone-axis data obtained for the unknown fibre, since non-amphibole minerals in some orientations may yield similar patterns consistent with amphibole structures.

The zone-axis ED interpretation shall include all minerals previously selected from the mineral data file as being chemically compatible with the EDXA data. This procedure will usually shorten the list of minerals for which solutions have been found. A second set of zone-axis data from another pattern obtained on the same fibre can then be processed, either as further confirmation of the identification, or to attempt elimination of an ambiguity. In addition, the angle measured between the orientations of the two zone-axes can be checked for consistency with the structures of the minerals. Caution should be exercised in rationalizing the inter-zone-axis angle, since if the fibre contains $\vec{c}$-axis twinning, the two zone-axis ED patterns may originate from the separate twin crystals. In practice, the full identification procedure will normally be applied to very few fibres, unless precise identification of all fibres is required for a particular reason.

D.4 Fibre classification categories

It is not always possible to proceed to a definitive identification of a fibre; this may be due to instrumental limitations or to the actual nature of the fibre. In many analyses, a definitive identification of each fibre may not actually be necessary if there is other knowledge available about the sample, or if the concentration is below a level of interest. The analytical procedure shall therefore take into account both instrumental limitations and varied analytical requirements. Accordingly, a system for fibre classification is used to permit accurate recording of data. The classifications are shown in tables D.1 and D.2, and are directed towards identification of chrysotile and amphibole respectively. Fibres shall be reported in these categories.

The general principle to be followed in this analytical procedure is first to define the most specific fibre classification which is to be attempted, or the "level" of analysis to be conducted. Then, for each fibre examined, record the classification which is actually achieved. Depending on the intended use of the results, criteria for acceptance of fibres as "identified" can then be established at any time after completion of the analysis.

In an unknown sample, chrysotile will be regarded as confirmed only if a recorded, calibrated ED pattern from one fibre in the CD categories is obtained, or if measurements of the ED pattern are recorded at the instrument. Amphibole will be regarded as confirmed only by obtaining recorded data which indicates exclusively the presence of amphiboles for fibres classified in the AZQ, AZZ or AZZQ categories.

Table D.1 — Classification of fibres with tubular morphology

Category	Description
TM	Tubular Morphology, not sufficiently characteristic for classification as chrysotile
CM	Characteristic Chrysotile Morphology
CD	Chrysotile SAED pattern
CQ	Chrysotile composition by Quantitative EDXA
CMQ	Chrysotile Morphology and composition by Quantitative EDXA
CDQ	Chrysotile SAED pattern and composition by Quantitative EDXA
NAM	Non-Asbestos Mineral

Table D.2 — Classification of fibres without tubular morphology

Category	Description
UF	Unidentified Fibre
AD	Amphibole by random orientation SAED (shows layer pattern of 0,53 nm spacing)
AX	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by Quantitative EDXA
AZ	Amphibole by one Zone-axis SAED pattern
ADQ	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	Amphibole by one Zone-axis SAED pattern and Quantitative EDXA
AZZ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle
AZZQ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle, and Quantitative EDXA
NAM	Non-Asbestos Mineral

D.4.1 Procedure for classification of fibres with tubular morphology suspected to be chrysotile

Occasionally, fibres are encountered which have tubular morphology similar to that of chrysotile, but which cannot be characterized further either by ED or EDXA. They may be non-crystalline, in which case ED techniques are not useful, or they may be in a position on the grid which does not permit an EDXA spectrum to be obtained. Alternatively, the fibre may be of organic origin, but the morphology and composition may not be sufficiently definitive enough to be disregarded. Accordingly, there is a requirement to record each fibre, and to specify how confidently each fibre can be identified. Classification of fibres will meet with various degrees of success. Figure D.2 shows the classification procedure to be used for fibres which display any tubular morphology. The chart is self explanatory, and every fibre is either rejected as a non-asbestos mineral (NAM), or classified in some way which by some later criterion could still contribute to the chrysotile fibre count.

Morphology is the first consideration, and if this is not similar to that usually seen in chrysotile standard samples, designate the initial classification as TM. Regardless of the doubtful morphology, examine the fibre by ED and EDXA methods according to figure D.2. Where the morphology is more definitive, it may be possible to classify the fibre as having chrysotile morphology (CM).

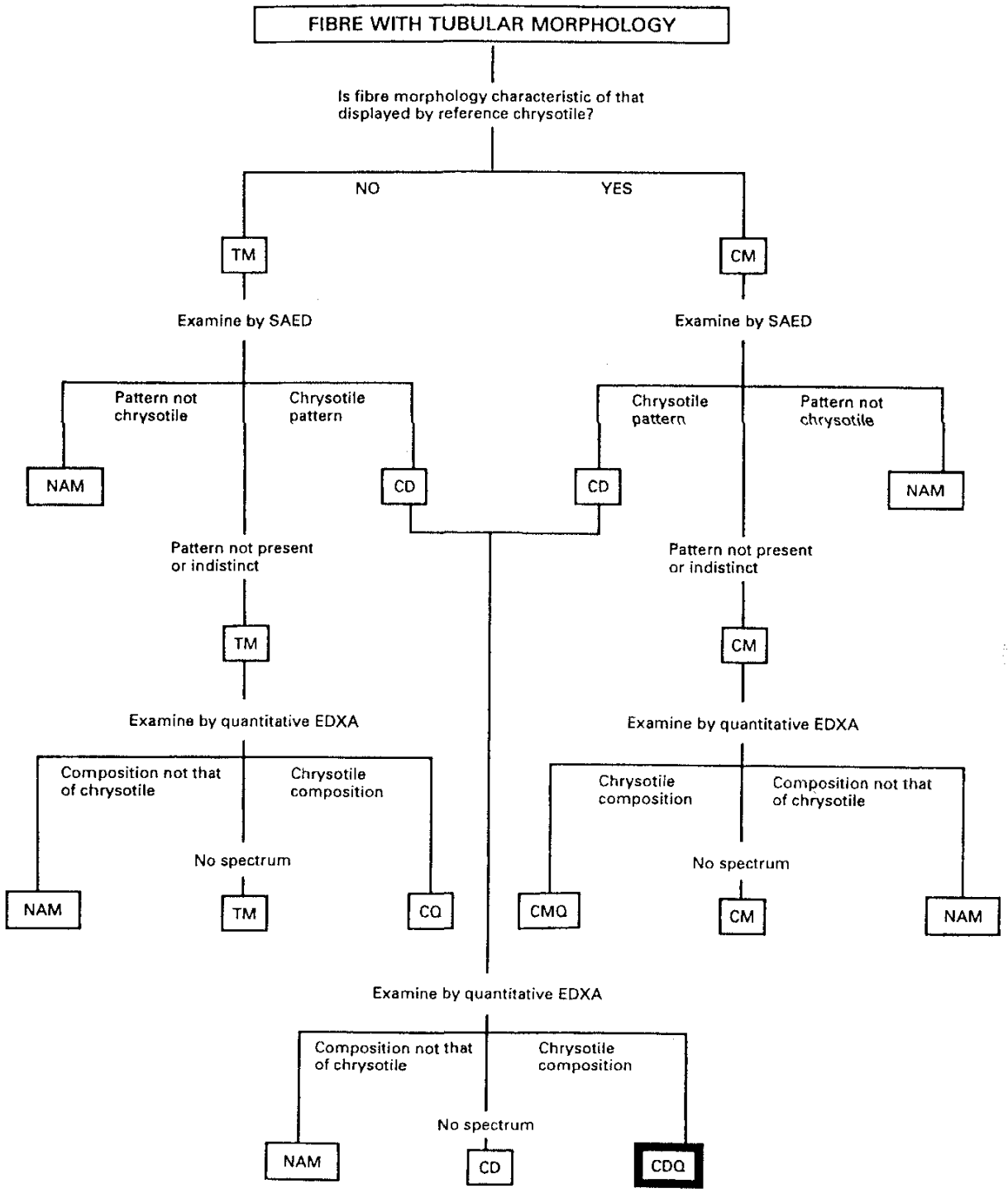


Figure D.2 — Classification chart for fibre with tubular morphology

For classification as CM, the morphological characteristics required are the following:

a) the individual fibrils should have high aspect ratios exceeding 5/1, and be about 30 nm to 40 nm in diameter;

b) the electron scattering power of the fibre at 60 kV to 100 kV accelerating potential should be sufficiently low for the internal structure to be visible;

c) there should be some evidence of an internal structure suggesting a tubular appearance similar to that shown by reference UICC chrysotile, which may degrade in the electron beam.

Examine every fibre having these morphological characteristics by the ED technique, and classify as chrysotile by ED (CD) only those which give diffraction patterns with the precise characteristics shown in figure D.3. The relevant features in this pattern for identification of chrysotile are as follows:

a) the (002) reflections should be examined to determine that they correspond closely to a spacing of 0,73 nm;

b) the layer line repeat distance should correspond to 0,53 nm;

c) there should be "streaking" of the (110) and (130) reflections.

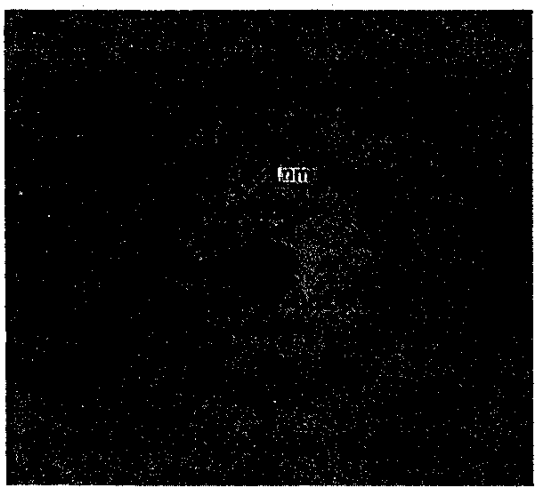


Figure D.3 — Chrysotile SAED pattern

Using the millimetre calibrations on the TEM viewing screen, these observations can readily be made at the instrument. If documentary proof of fibre identification is required, record a TEM micrograph of at least one representative fibre, and record its ED pattern on a separate film or plate. This film or plate shall also carry calibration rings from a known polycrystalline substance such as gold. This calibrated pattern is the only documentary proof that the particular fibre is chrysotile, and not some other tubular or scrolled species such as halloysite, palygorskite, talc or vermiculite. The proportion of fibres which can be successfully identified as chrysotile by ED is variable, and to some extent dependent on both the instrument and the procedures of the operator. The fibres that fail to yield an identifiable ED pattern will remain in the TM or CM categories unless they are examined by EDXA.

In the EDXA analysis of chrysotile there are only two elements which are relevant. For fibre classification, the EDXA analysis shall be quantitative. If the spectrum displays prominent peaks from magnesium and silicon, with their areas in the appropriate ratio, and with only minor peaks from other elements, classify the fibre as chrysotile by quantitative EDXA, in the categories CQ, CMQ, or CDQ, as appropriate.

D.4.2 Procedure for classification of fibres without tubular morphology, suspected to be amphibole

Every particle without tubular morphology and which is not obviously of biological origin, with an aspect ratio of 5/1 or greater, and having parallel or stepped sides, shall be considered as a suspected amphibole fibre. Further examination of the fibre by ED and EDXA techniques will meet with a variable degree of success, depending on the nature of the fibre and on a number of instrumental limitations. It will not be possible to identify every fibre completely, even if time and cost are of no concern. Moreover, confirmation of the presence of amphibole can be achieved only by quantitative interpretation of zone-axis ED patterns, a very time-consuming procedure. Accordingly, for routine samples from unknown sources, this analytical procedure limits the requirement for zone-axis ED work to a minimum of one fibre representative of each compositional class reported. In some samples, it may be necessary to identify more fibres by the zone-axis technique. When analysing samples from well-characterized sources, the cost of identification by zone-axis methods may not be justified.

The 0,53 nm layer spacing of the random orientation ED pattern is not by itself diagnostic for amphibole, However, the presence of $\vec{c}$-axis twinning in many fi-

bres leads to contributions to the layers in the patterns by several individual parallel crystals of different axial orientations. This apparently random positioning of the spots along the layer lines, if also associated with a high fibre aspect ratio, is a characteristic of amphibole asbestos, and thus has some limited diagnostic value. If a pattern of this type is not obtained, the identity of the fibre is still ambiguous, since the absence of a recognizable pattern may be a consequence of an unsuitable orientation relative to the electron beam, or the fibre may be some other mineral species.

Figure D.4 shows the fibre classification chart to be used for suspected amphibole fibres. This chart shows all the classification paths possible in analysis of a suspected amphibole fibre, when examined systematically by ED and EDXA. Two routes are possible, depending on whether an attempt to obtain an EDXA spectrum or a random orientation ED pattern is made first. The normal procedure for analysis of a sample of unknown origin will be to examine the fibre by random orientation ED, qualitative EDXA, quantitative EDXA, and zone-axis ED, in this sequence. The final fibre classification assigned will be defined either by successful analysis at the maximum required level, or by the instrumental limitations. Any instrumental limitations which affect the quality of the results sh be noted. Record the maximum classification achieved for each fibre on the counting sheet in the appropriate column. The various classification categories can then be combined later in any desired way for calculation of the fibre concentration. The complete record of the results obtained when attempting to identify each fibre can also be used to re-assess the data if necessary.

In the unknown sample, zone-axis analysis will be required if the presence of amphibole is to be unequivocally confirmed. For this level of analysis, attempt to raise the classification of every suspected amphibole fibre to the ADQ category by inspection of the random orientation ED pattern and the EDXA spectrum. In addition, examine at least one fibre from each type of suspected amphibole found by zone-axis methods to confirm their identification. In most cases, because information exists about possible sources of asbestos in close proximity to the air sampling location, some degree of ambiguity of identification can be accepted. Lower levels of analysis can therefore be accepted for these situations.

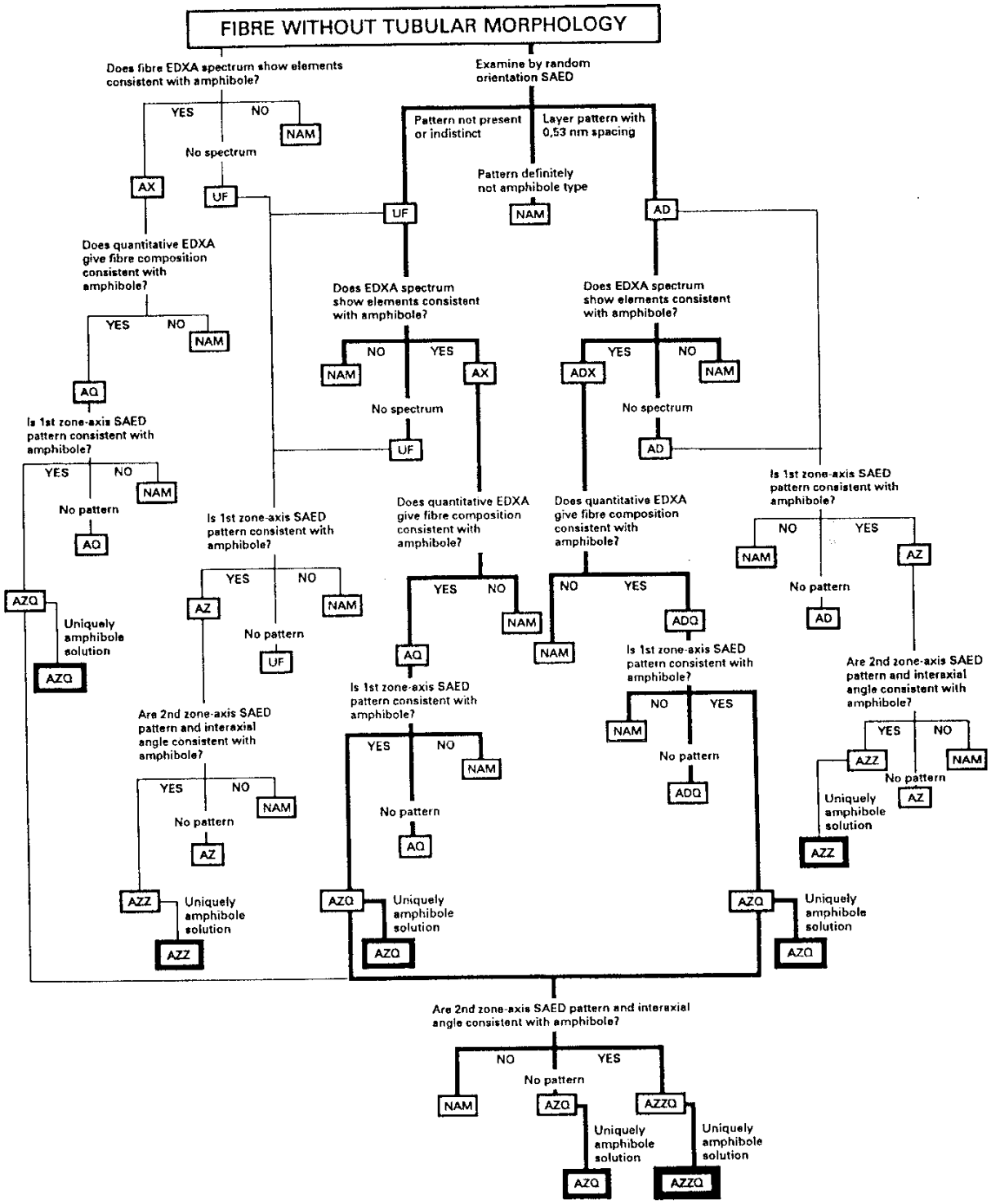


Figure D.4 — Classification chart for fibre without tubular morphology

Annex E
(normative)

Determination of the concentrations of asbestos fibres and bundles longer than 5 μm, and PCM equivalent asbestos fibres

In order to provide increased statistical precision and improved analytical sensitivity for those asbestos fibres and bundles longer than 5 μm, it may be decided to perform additional fibre counting at a lower magnification, taking account only into fibres and bundles within this dimensional range. The result shall be specified as "number of asbestos fibres and bundles longer than 5 μm". For this examination, use a magnification of approximately × 10 000, and continue to assign a morphological code to each structure according to the procedures specified in annex C. Record fibres and bundles only if their lengths exceed 5 μm. Record cluster and matrix components only if their lengths exceed 5 μm.

It may also be decided to provide increased statistical precision and improved analytical sensitivity for fibrous structures longer than 5 μm, with diameters between 0,2 μm and 3,0 μm, which have historically been the basis of risk estimation in the occupational environment (PCM equivalent asbestos fibres). Use a magnification of approximately × 5 000 for this extended fibre count. The result shall be specified as "number of PCM equivalent asbestos fibres". Asbestos structures within this dimensional range do not necessarily incorporate asbestos fibres or bundles longer than 5 μm.

Continue the extended sample examination until 100 asbestos structures have been counted, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity calculated according to table 1. The grid openings examined shall be divided approximately equally between a minimum of two specimen grids.

NOTES

17 The specimen area corresponding to the area of filter examined in the PCM fibre counting methods is 0,785 mm^2, and is equivalent to approximately 100 grid openings of a 200 mesh grid.

18 Some National Standards require that asbestos fibres longer than 2,5 μm, with diameters between 0,2 μm and 3,0 μm be counted. Use a magnification of × 5 000 for counting fibres within these dimensional ranges.

19 The minimum aspect ratio for definition of a fibre in PCM fibre counting methods and in some National Standards is 3/1. Use of a 3/1 aspect ratio is permitted in this International Standard, if this aspect ratio is mentioned in the test report.

The test reports shall include all of the items listed in clause 11.

Annex F
(normative)

Calculation of results

F.1 General

The results should be calculated using the procedures specified below. The results can be conveniently calculated using a computer programme.

F.2 Test for uniformity of distribution of fibrous structures on TEM grids

A chek shall be made using the chi-square test, to determine whether the asbestos structures found on individual grid openings are randomly and uniformly distributed among the grid openings. If the total number found in k grid openings is n, and the areas of the k individual frid openings are designated A_1 to A_k, then the total area of TEM specimen examined is

$$A = \sum_{i=1}^{i=k} A_i$$

The fraction of the total area examined which is represented by the individual grid opening area, p_i, is given by A_i/A. If the structures are randomly and uniformly dispersed over the k grid openings examined, the expected number of structures falling in one grid opening with area A_i is np_i. If the observed number of structures found on that grid opening is n_i, then

$$x^2 = \sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

This value shall be compared with significance points of the chi-square distribution, having $(k - 1)$ degrees of freedom. Significance levels lower than 0,1 % may be cause for the sample analysis to be rejected, since this correspond to a very inhomogeneous deposit. If the structure count fails this test, the precision of the result will be uncertain, and if new air samples cannot be collected, additional grid openings may be examined or the sample may be prepared by an indirect method.

F.3 Calculation of the analytical sensitivity

Calculate the required analytical sensitivity S, expressed in number of structures per litre, using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

A_f is the area, in square millimetres, of sample collection filter;

A_g is the area, in square millimetres, of TEM specimen grid opening;

k is the number of grid openings examined;

V is the volume of air sampled, in litres.

F.4 Calculation of the mean and confidence interval of the structure concentration

In the structure count made according to this International Standard, a number of grid openings have been sampled from a population of grid openings, and it is required to determine the mean grid opening structure count for the population on the basis of this small sample. The interval about the sample mean which, with 95 % confidence, contains the population mean, is also required.

F.4.1 Calculation of the mean structure concentration

Calculate the mean structure concentration C, expressed in number of structures per litre, using the following equation:

$$C = Sn$$

where

S is the analytical sensitivity, expressed in number of structures per litre;

n is the total number of structures found on all grid openings examined.

F.4.2 Calculation of confidence intervals

The distribution of structures on the grid openings should theoretically approximate to a Poisson distribution. Because of fibre aggregation and size-dependent identification effects, the actual structure counting data often does not conform to the Poisson distribution, particularly at high structure counts. An assumption that the structure counting data are distributed according to the Poisson distribution can therefore lead to confidence intervals narrower than are justified by the data. Moreover, if the Poisson distribution is assumed, the variance is related only to the total number of structures counted. Thus, a particular structure count conducted on one grid opening is considered to have the same confidence interval as that for the same number of structures found on many grid openings. However, the area of sample actually counted is very small in relation to the total area of the filter, and for this reason, structures shall be counted on a minimum of four grid openings taken from different areas of the filter in order to ensure that a representative evaluation of the deposit is made.

At high structure counts, where there are adequate numbers of structures per grid opening to allow a sample estimate of the variance to be made, the distribution can be approximated to a Gaussian, with independent values for the mean and variance. Where the sample estimate of variance exceeds that implicit in the Poissonian assumption, use of Gaussian statistics with the variance defined by the actual data is the most conservative approach to calculation of confidence intervals.

At low structure counts, it is not possible to obtain a reliable sample estimate of the variance, and the distribution also becomes asymmetric but not necessarily Poissonian. For 30 structures and below, the distribution becomes asymmetric enough for the fit to a Gaussian to no longer be a reasonable one, and estimates of sample variance are unreliable. Accordingly, for counts below 31 structures, the assumption of a Poisson distribution shall be made for calculation of the confidence intervals.

F.4.3 Example of calculation of Poissonian 95 % confidence intervals

For total structure counts less than 4, the lower 95 % confidence limit corresponds to less than 1 structure. Therefore, it is not meaningful to quote lower confidence interval points for structure counts of less than 4, and the result shall be recorded as "less than" the corresponding one-sided upper 95 % confidence limit of the Poisson distribution, as follows:

0 structure ≙ 2,99 times the analytical sensitivity

1 structure ≙ 4,74 times the analytical sensitivity

2 structures ≙ 6,30 times the analytical sensitivity

3 structures ≙ 7,75 times the analytical sensitivity

For total counts exceeding 4, the 95 % confidence interval shall be calculated using the values shown in table F.1. Table F.1 gives the upper and lower limits of the two-sided Poissonian 95 % confidence interval for structure counts up to 470.

F.4.4 Example of calculation of Gaussian 95 % confidence intervals

Calculate the sample estimate of variance s^2 using the following equation:

$$s^2 = \frac{\sum_{i=1}^{i=k} (n_i - np_i)^2}{(k-1)}$$

where

n_i is the number of structures on the ith grid opening;

n is the total number of structures found in k grid openings;

p_i is the fraction of the total area examined represented by the ith grid opening;

k is the number of grid openings examined.

If the mean value of the structure count is calculated to be n, the upper and lower values of the Gaussian 95 % confidence interval are given respectively by

$$L_u = \frac{n}{k} + \frac{ts}{\sqrt{k}}$$

and

$$L_l = \frac{n}{k} - \frac{ts}{\sqrt{k}}$$

where

L_u is the upper 95 % confidence limit;

L_l is the lower 95 % confidence limit;

n is the total number of structures in all grid openings examined;

t is the value of Student's test (probability 0,975) for $(k - 1)$ degrees of freedom;

s is the standard deviation (square root of sample estimate of variance);

k is the number of grid openings examined.

F.4.5 Summary of procedure for calculation of results

In summary, structure counting data shall be calculated as follows:

No structures detected

The structure concentration shall be reported as less than the concentration equivalent ot the one-sided upper 95 % confidence limit of the Poisson distribution. This is equal to 2,99 times the analytical sensitivity.

From 1 to 3 structures

When 1 to 3 structures are counted, the result shall be reported as less than the corresponding one-sided upper 95 % confidence limit for the Poisson distribution. These are

1 structure ≙ 4,74 times the analytical sensitivity

2 structures ≙ 6,30 times the analytical sensitivity

3 structures ≙ 7,75 times the analytical sensitivity

From 4 to 30 structures

The mean structure concentration and the 95 % confidence intervals shall be reported on the basis of the Poissonian assumption, using the values shown in table F.1.

More than 30 structures

When more 30 structures are counted, both the Gaussian 95 % confidence interval and the Poissonian 95 % confidence interval shall be calculated. The larger of these two intervals shall be used to express the precision of the structure concentration. When the Gaussian 95 % confidence interval is selected for data reporting, the Poissonian 95 % confidence interval shall also be mentioned.

F.5 Calculation of structure length, width, and aspect ratio distributions

The distributions all approximate to logarithmic-normal, and therefore the size range intervals for calculation of the distribution shall be spaced logarithmically. The other characteristics required for the choice of size intervals are that they should allow for a sufficient number of size classes, while still retaining a statistically valid number of structures in each class. Interpretation is also facilitated if each size class repeats at 10 intervals, and if 5 µm is a size class boundary. A ratio from one class to the next of 1,468 satisfies all of these requirements and this value shall be used. The distributions, being approximately logarithmic-normal, when presented graphically, shall be plotted using a logarithmic ordinate scale and a Gaussian abscissa.

F.5.1 Calculation of structure length cumulative number distribution

This distribution allows the fraction of the total number of structures either shorter or longer than a given length to be determined. It is calculated using the following equation:

$$C(P)_k = \frac{\sum_{i=1}^{i=k} n_i}{\sum_{i=1}^{i=P} n_i} \times 100$$

where

$C(P)_k$ is the cumulative number percentage of structures which have lengths less than the upper bound of the kth class;

n_i is the number of structures in the ith length class;

P is the total number of length classes.

F.5.2 Calculation of structure width cumulative number distribution

This distribution allows the fraction of the total number of structures either narrower or wider than a given width to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure widths.

F.5.3 Calculation of structure aspect ratio cumulative number distribution

This distribution allows the fraction of the total number of structures which have aspect ratios either smaller or larger than a given aspect ratio to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure aspect ratios.

Table F.1 — Upper and lower limits of the Poissonian 95 % confidence interval of a count

Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit
0	0	3,689[1)]	46	33,678	61,358	92	74,164	112,83
1	0,025	5,572	47	34,534	62,501	93	75,061	113,94
2	0,242	7,225	48	35,392	63,642	94	75,959	115,04
3	0,619	8,767	49	36,251	64,781	95	76,858	116,14
4	1,090	10,242	50	37,112	65,919	96	77,757	117,24
5	1,624	11,669	51	37,973	67,056	97	78,657	118,34
6	2,202	13,060	52	38,837	68,192	98	79,557	119,44
7	2,814	14,423	53	39,701	69,326	99	80,458	120,53
8	3,454	15,764	54	40,567	70,459	100	81,360	121,66
9	4,115	17,085	55	41,433	71,591	110	90,400	132,61
10	4,795	18,391	56	42,301	72,721	120	99,490	143,52
11	5,491	19,683	57	43,171	73,851	130	108,61	154,39
12	6,201	20,962	58	44,041	74,979	140	117,77	165,23
13	6,922	22,231	59	44,912	76,106	150	126,96	176,04
14	7,654	23,490	60	45,785	77,232	160	136,17	186,83
15	8,396	24,741	61	46,658	78,357	170	145,41	197,59
16	9,146	25,983	62	47,533	79,482	180	154,66	208,33
17	9,904	27,219	63	48,409	80,605	190	163,94	219,05
18	10,668	28,448	64	49,286	81,727	200	173,24	229,75
19	11,440	29,671	65	50,164	82,848	210	182,56	240,43
20	12,217	30,889	66	51,042	83,969	220	191,89	251,10
21	13,00	32,101	67	51,922	85,088	230	201,24	261,75
22	13,788	33,309	68	52,803	86,207	240	210,60	272,39
23	14,581	34,512	69	53,685	87,324	250	219,97	283,01
24	15,378	35,711	70	54,567	88,441	260	229,36	293,62
25	16,178	36,905	71	55,451	89,557	270	238,75	304,23
26	16,983	38,097	72	56,335	90,673	280	248,16	314,82
27	17,793	39,284	73	57,220	91,787	290	257,58	325,39
28	18,606	40,468	74	58,106	92,901	300	267,01	335,96
29	19,422	41,649	75	58,993	94,014	310	276,45	346,52
30	20,241	42,827	76	59,880	95,126	320	285,90	357,08
31	21,063	44,002	77	60,768	96,237	330	295,36	367,62
32	21,888	45,175	78	61,657	97,348	340	304,82	378,15
33	22,715	46,345	79	62,547	98,458	350	314,29	388,68
34	23,545	47,512	80	63,437	99,567	360	323,77	399,20
35	24,378	48,677	81	64,328	100,68	370	333,26	409,71
36	25,213	49,840	82	65,219	101,79	380	342,75	420,22
37	26,050	51,000	83	66,111	102,90	390	352,25	430,72
38	26,890	52,158	84	67,003	104,00	400	361,76	441,21
39	27,732	53,315	85	67,897	105,11	410	371,27	451,69
40	28,575	54,469	86	68,790	106,21	420	380,79	462,18
41	29,421	55,622	87	69,684	107,32	430	390,32	472,65
42	30,269	56,772	88	70,579	108,42	440	399,85	483,12
43	31,119	57,921	89	71,474	109,53	450	409,38	493,58
44	31,970	59,068	90	72,370	110,63	460	418,92	504,04
45	32,823	60,214	91	73,267	111,73	470	428,47	514,50

1) The one-sided upper 95 % confidence limit for 0 structures is 2,99.

Annex G
(informative)

Strategies for collection of air samples

G.1 General

An important part of the sampling strategy is a statement of the purpose of the sampling programme. A sufficient number of samples should be collected so that the site is well characterized to the precision and accuracy desired, and also ensure that sample filters appropriately loaded for TEM analysis are obtained from all of the sampling locations.

G.2 Air sample collection in the outdoors environment

Weather conditions restrict the ability to collect satisfactory air samples in the outdoors environment, and whenever possible, sampling should be carried out in low-wind, low-humidity conditions. Detailed records of the weather conditions, windspeed and direction during the sampling period should be made. All available information concerning local topography, and the types and positions of sources should be recorded.

Sequential multipoint sampling is necessary to provide adequate characterization of complex sites and sources. It is recommended that multiple samples are taken upwind and downwind of the site, with a minimum of two samples in the downwind position expected to experience the maximum airborne concentration. The locations of the samplers should be carefully recorded.

G.3 Air sample collection inside buildings

Air samples are often collected inside buildings in which asbestos-containing construction materials are present, in order to determine whether these materials contribute to the asbestos fibre concentration in the building atmosphere. The optimum positions for collection of air samples can only be determined after a complete survey of the building to establish air movement patterns. Multiple samples should be collected in the area where asbestos building materials are present, and control samples should be collected in an adjacent area where no airborne asbestos fibres would be expected. The intakes for air conditioning systems are frequently used as the collection locations for control samples. Whenever possible, static samples should be taken over a period exceeding 4 h during normal activity in the building, at face velocities of between 4 cm/s and 25 cm/s.

Annex H
(informative)

Methods for removal of gypsum fibres

It is common to find fibres of calcium sulfate (gypsum) in airborne particulates collected in buildings and urban environments, and particularly in samples collected where demolition or construction work is in progress. The fibres are readily released when plasters and cement products are disturbed. In some circumstances, particles of calcite or dolomite collected on an air filter can react with atmospheric sulfur dioxide, to form long fibres of gypsum. Gypsum fibres can give rise to high fibre counts by both optical and electron microscopy. The gypsum fibres are often 2 μm to 6 μm long, with aspect ratios greater than 10/1. Sometimes, these fibres appear similar to amphibole asbestos fibres, and in some samples they can be morphologically very similar to chrysotile. In the TEM, the larger fibres have high contrast and at high magnification often exhibit a characteristic mottled appearance which changes under electron beam irradiation. Some gypsum fibres, however, are not easily discriminated from asbestos without examination by EDXA. TEM specimens which contain many such gypsum fibres require an extended examination time in the TEM, because it is necessary to examine each of these fibres by EDXA before it can be rejected.

It is possible to remove gypsum fibres selectively by water extraction. A Jaffe washer (7.3.7), or a condensation washer (7.3.8), should be prepared, but using a water (6.1) as the solvent. The TEM specimens, which have been previously prepared and initially examined in the TEM, should be placed in the washer to allow dissolution of the fibres. If a Jaffe washer is used, the treatment time can be reduced by heating the washer to 90 °C to 100 °C for a few minutes. If a condensation washer is used, the gypsum fibres will be dissolved by treatment for approximately 10 min. The effect of this treatment is to remove the gypsum fibres, leaving carbon replicas (7.3.11) which are readily distinguished from asbestos fibres.

NOTE 20 This procedure should be used only when examination of the untreated TEM specimen grids shows the gypsum fibres to be isolated from any asbestos fibres present. Losses of asbestos fibres may occur if matrices of gypsum and asbestos are exposed to this procedure.

Annex J
(informative)

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ICS 13.040.20

Descriptors: air, quality, air pollution, tests, determination, particle density (concentration), asbestos, microscopic analysis.

Price based on 51 pages

ASBESTOS and OTHER FIBERS by PCM 7400

FORMULA: Various MW: Various CAS: see Synonyms RTECS: Various

METHOD: 7400, Issue 2 **EVALUATION: FULL** **Issue 1: Rev. 3 on 15 May 1989**
Issue 2: 15 August 1994

OSHA: 0.1 asbestos fiber (> 5 µm long)/cc; 1 f/cc, 30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 µm long), 400 L; carcinogen
ACGIH: 0.2 f/cc crocidolite; 0.5 f/cc amosite; 2 f/cc chrysotile and other asbestos; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4]; refractory ceramic fibers [142844-00-6]; fibrous glass

SAMPLING

SAMPLER: FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm; conductive cowl on cassette)

FLOW RATE*: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)

*Adjust to give 100 to 1300 fiber/mm^2

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION ($\hat{S}_{rT}$): 0.115 to 0.13 [1]

ACCURACY: see EVALUATION OF METHOD

MEASUREMENT

TECHNIQUE: LIGHT MICROSCOPY, PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone - collapse/triacetin - immersion method [2]

COUNTING RULES: described in previous version of this method as "A" rules [1,3]

EQUIPMENT:
1. positive phase-contrast microscope
2. Walton-Beckett graticule (100-µm field of view) Type G-22
3. phase-shift test slide (HSE/NPL)

CALIBRATION: HSE/NPL test slide

RANGE: 100 to 1300 fibers/mm^2 filter area

ESTIMATED LOD: 7 fibers/mm^2 filter area

PRECISION ($\bar{S}_r$): 0.10 to 0.12 [1]; see EVALUATION OF METHOD

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fibers < ca. 0.25 µm diameter will not be detected by this method [4]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

INTERFERENCES: If the method is used to detect a specific type of fiber, any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This revision replaces Method 7400, Revision #3 (dated 5/15/89).

REAGENTS:

1. Acetone,* reagent grade.
2. Triacetin (glycerol triacetate), reagent grade.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically conductive extension cowl and cellulose ester filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is ≥ 5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.
   NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.
   NOTE 3: Use 0.8-µm pore size filters for personal sampling. The 0.45-µm filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.
   NOTE 4: Other cassettes have been proposed that exhibit improved uniformity of fiber deposit on the filter surface, e.g., bellmouthed sampler (Envirometrics, Charleston, SC). These may be used if shown to give measured concentrations equivalent to sampler indicated above for the application.
2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow-rate requirements (see step 4 for flow rate), with flexible connecting tubing.
3. Wire, multi-stranded, 22-gauge; 1″ hose clamp to attach wire to cassette.
4. Tape, shrink- or adhesive-.
5. Slides, glass, frosted-end, pre-cleaned, 25- × 75-mm.
6. Cover slips, 22- × 22-mm, No. 1½, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

EQUIPMENT (continued):

10. Acetone flash vaporization system for clearing filters on glass slides (see ref. [5] for specifications or see manufacturer's instructions for equivalent devices).
11. Micropipets or syringes, 5-µL and 100- to 500-µL.
12. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10× eyepiece, and 40 to 45× phase objective (total magnification ca. 400×); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100-µm diameter circular field (area = 0.00785 mm^2) at the specimen plane (Type G-22). Available from Optometrics USA, P.O. Box 699, Ayer, MA 01432 [phone (508)-772-1700], and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
    NOTE: The graticule is custom-made for each microscope. (see APPENDIX A for the custom-ordering procedure).
14. HSE/NPL phase contrast test slide, Mark II. Available from Optometrics USA (address above).
15. Telescope, ocular phase-ring centering.
16. Stage micrometer (0.01-mm divisions).

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.
    NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E, of 100 to 1300 fibers/mm^2 (3.85×10^4 to 5×10^5 fibers per 25-mm filter with effective

collection area A_c = 385 mm^2) for optimum accuracy. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \times E}{Q \times L \times 10^3}.$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [7]. Relatively large diameter fibers (>3 µm) may exhibit significant aspiration loss and inlet deposition. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If ≥50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min [3].

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤ 1.46. This method collapses the filter for easier focusing and produces permanent (1–10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400—revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7–9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9–11].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [2].
   NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
   a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.
      NOTE: Static electricity will usually keep the wedge on the slide.
   b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250 µL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the

acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 s for the filter to clear, remove pipet and slide from their ports.

CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce explosive acetone vapor concentrations.

c. Using the 5-µL micropipet, immediately place 3.0 to 3.5 µL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.

NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.

e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.

NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturer's instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.

    a. Each time a sample is examined, do the following:

        (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.

        (2) Focus on the particulate material to be examined.

        (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

    b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:

        (1) Center the HSE/NPL phase-contrast test slide under the phase objective.

        (2) Bring the blocks of grooved lines into focus in the graticule area.

        NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting, the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.

        (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Document the laboratory's precision for each counter for replicate fiber counts.

    a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer should maintain custody of the reference slides and should supply each counter with a minimum of one reference

slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter precision. Obtain separate values of relative standard deviation (S_r) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.

NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [9].

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm^2) exceeds $2.77XS_r'$ where X = average of the square roots of the two fiber counts (in fiber/mm^2) and $S_r' = S_r / 2$ where S_r is the intracounter relative standard deviation for the appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [13].

NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10- to 20-minute breaks from the microscope every one or two hours to limit fatigue [14]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.
15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program for asbestos and routinely exchange field samples with other laboratories to compare performance of counters.

MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.
17. Adjust the microscope (Step 10).

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm) [4].

18. Counting rules: (same as P&CAM 239 rules [1,10,11]: see examples in APPENDIX B).

a. Count any fiber longer than 5 µm which lies entirely within the graticule area.
   (1) Count only fibers longer than 5 µm. Measure length of curved fibers along the curve.
   (2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.

b. For fibers which cross the boundary of the graticule field:
   (1) Count as ½ fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.

(2) Do not count any fiber which crosses the graticule boundary more than once.
(3) Reject and do not count all other fibers.

c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.

19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 s per field is appropriate for accurate counting.

NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 µm), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].

NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.

NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].

NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/mm^2) is defined as the average count (fibers/field) divided by the field (graticule) area (mm^2/field).

CALCULATIONS AND REPORTING OF RESULTS

20. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the average fiber count per graticule field, F / n_f, minus the mean field blank count per graticule field, B / n_b, by the graticule field area, A_f (approx. 0.00785 mm^2):

$$E = \frac{(F/n_f - B/n_b)}{A_f}, \text{ fibers/mm}^2.$$

NOTE: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased." Other fiber counts outside the 100–1300 fiber/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm^2 for a 25-mm filter):

$$C = \frac{EA_c}{V \times 10^3}.$$

NOTE: Periodically check and adjust the value of A_c, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.
    NOTE: Precision depends on the total number of fibers counted [1,16]. Relative standard deviation is documented in references [1,15–17] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Figure 1).

EVALUATION OF METHOD:

Method Revisions:

This method is a revision of P&CAM 239 [10]. A summary of the revisions is as follows:

1. Sampling:
   The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].
2. Sample preparation technique:
   The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.
3. Measurement:
   a. The Walton-Beckett graticule standardizes the area observed [14,18,19].
   b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].
   c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm^2 filter area (a total of 78.5 fibers counted in 100 fields, each with field area = 0.00785 mm^2.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S_r in the range of 0.10 to 0.17 [21–23].

Interlaboratory Comparability:

An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [9]. The relative standard deviations (S_r) varied with sample type and laboratory. The ranges were:

Rules	Intralaboratory S_r	Interlaboratory S_r	Overall S_r
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)†	0.11 to 0.29	0.20 to 0.35	0.25

*Under AIA rules, only fibers having a diameter less than 3 µm are counted and fibers attached to particles larger than 3 µm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.

†See Appendix C.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S_r in the range 0.17 to 0.25 and an interlaboratory S_r of 0.45 [21]. This agrees well with other recent studies [9,14,16].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S_r. (NOTE: The following discussion does not include bias estimates and should not be taken to indicate that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an S_r that depends on the number, N, of fibers counted:

$$S_r = 1/N^{1/2}.$$

Thus S_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S_r found in a number of studies is greater than these theoretical numbers [17,19–21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [15] found this subjective component of intralaboratory S_r to be approximately 0.2 and estimated the overall S_r by the term:

$$\frac{[N + (0.2 \times N)^2]^{1/2}}{N}.$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 S_r and −1.5 S_r. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [14]. These results gave a subjective interlaboratory component of S_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [21]. This value falls slightly above the range of S_r (0.25 to 0.42 for 1984–85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [17].

A number of factors influence S_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is hoped that the laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the S_r.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [16].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 counting field area). If this same sample were counted by

a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the S_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory S_r is smaller, then it is more correct to use that smaller S_r. However, the estimated S_r of 0.45 is to be used in the absence of such information. Note also that it has been found that S_r can be higher for certain types of samples, such as asbestos cement [9].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., $0.16 + 2.13 \times 0.16 = 0.5$.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and −49% as the upper and lower confidence values of the mean for a 100-fiber count.

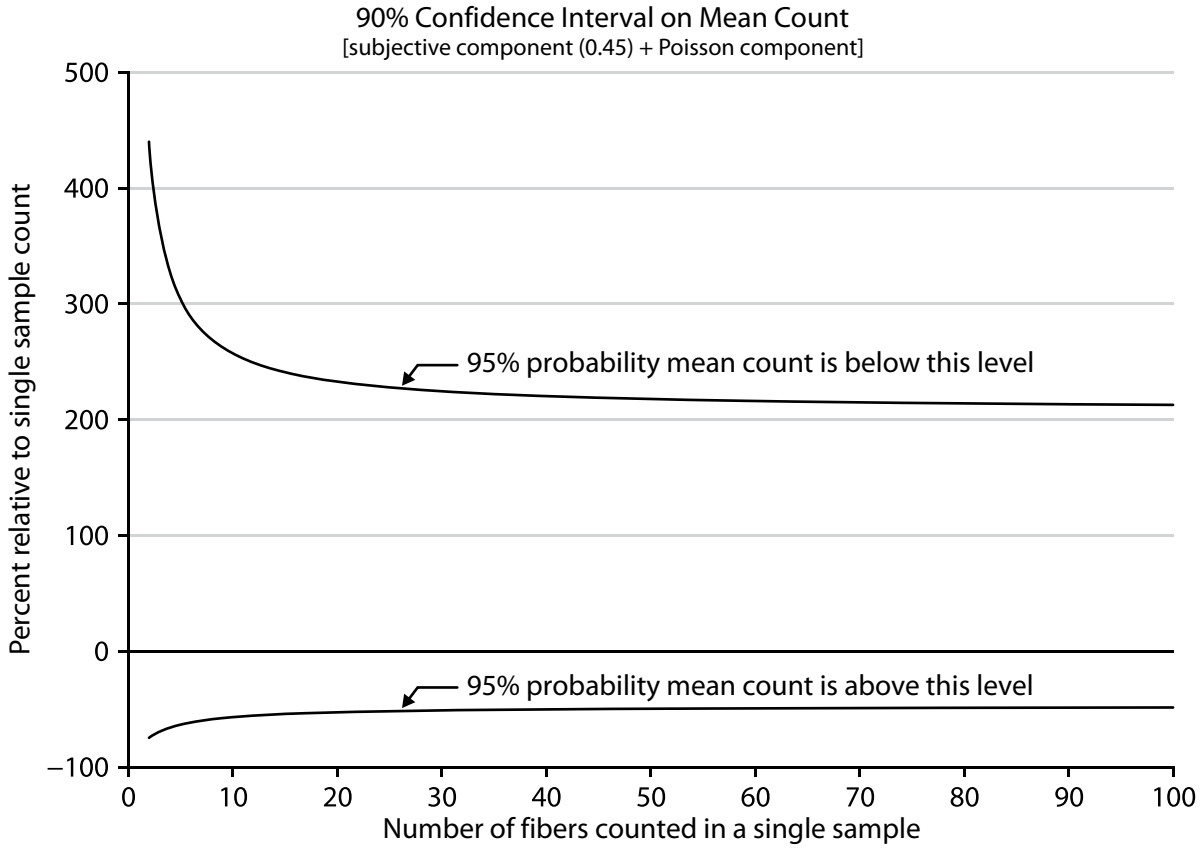

Figure 1. Interlaboratory precision of fiber counts.

The curves in Figure 1 are defined by the following equations:

$$U_{CL} = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25S_r^2)X^2]^{½}}{2(1 - 2.25S_r^2)} \text{ and}$$

$$L_{CL} = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4S_r^2)X^2]^{½}}{2(1 - 4S_r^2)},$$

where S_r = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory S_r when approximately 100 fibers are counted,
X = total fibers counted on sample,
L_{CL} = lower 95% confidence limit, and
U_{CL} = upper 95% confidence limit.

Note that the range between these two limits represents 90% of the total range.

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APPENDIX A. CALIBRATION OF THE WALTON-BECKETT GRATICULE

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (*D*) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45× phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_o} \times D.$$

   Example: If L_o = 112 µm, L_a = 4.5 mm, and D = 100 µm, then d_c = 4.02 mm.
8. Check the field diameter, *D* (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 mm^2 to 0.00817 mm^2).

APPENDIX B. COMPARISON OF COUNTING RULES

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. The rules will be discussed as they apply to the labeled objects in the figure.

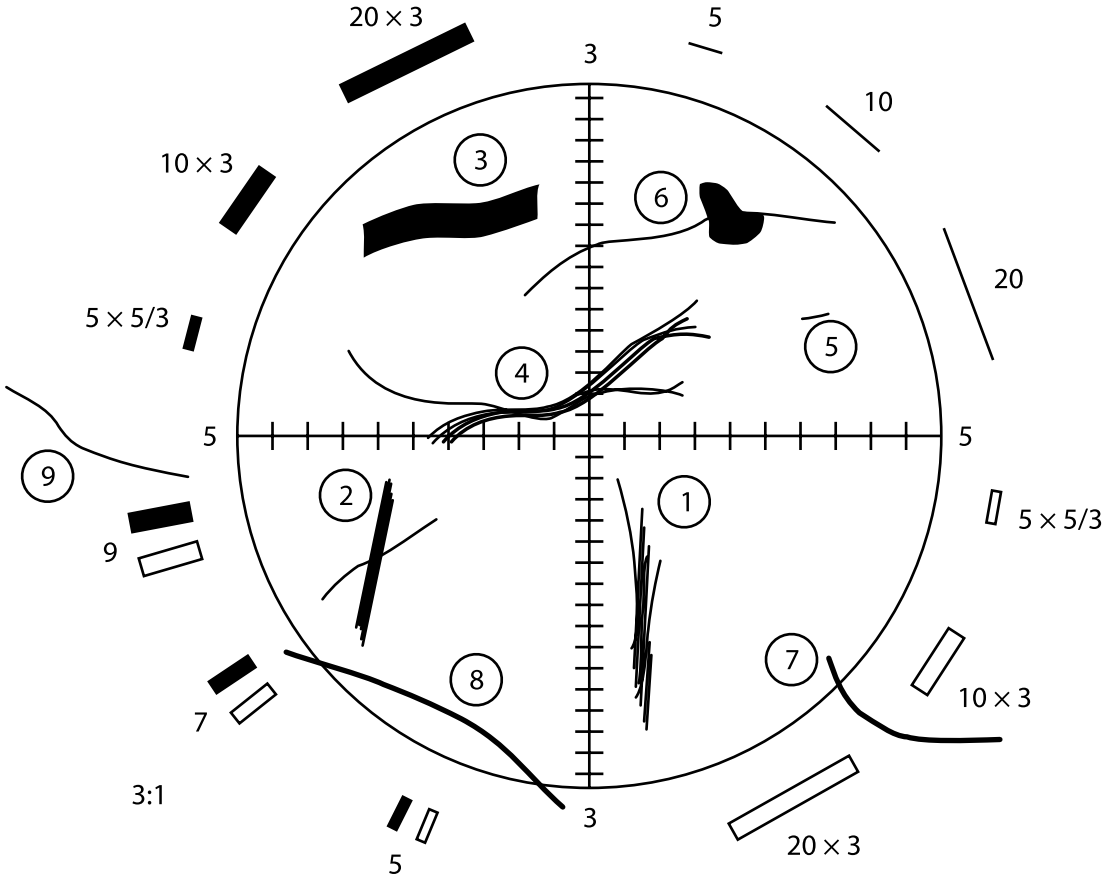

Figure 2. Walton-Beckett graticule with fibers.

These rules are sometimes referred to as the "A" rules:

Object	Count	Discussion
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle, the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fibers	If fibers meeting the length and aspect ratio criteria (length >5 µm and length-to-width ratio > 3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 µm), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is ≤ 5 µm long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	½ fiber	A fiber which crosses into the graticule area one time is counted as ½ fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.

APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules given below (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [24], and the NIOSH fibrous glass criteria document method [25]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" Counting Rules

1. Count only *ends* of fibers. Each fiber must be longer than 5 µm and less than 3 µm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 µm in diameter.

5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION

	Fiber density on filter*		Fiber concentration in air, f/cc	
	Fibers per 100 fields	Fibers/mm^2	400-L air sample	1000-L air sample
	200	255	0.25	0.10
	100	127	0.125	0.05
LOQ	80.0	102	0.10	0.04
	50	64	0.0625	0.025
	25	32	0.03	0.0125
	20	25	0.025	0.010
	10	12.7	0.0125	0.005
	8	10.2	0.010	0.004
LOD	5.5	7	0.00675	0.0027

*Assumes 385 mm^2 effective filter collection area, and field area = 0.00785 mm^2, for relatively "clean" (little particulate aside from fibers) filters.

ASBESTOS by TEM 7402

FORMULA: Various MW: Various CAS: Various RTECS: Various

METHOD: 7402 **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

OSHA : 0.1 asbestos fibers (>5 µm long)/cc; 1 f/cc/30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anistropic

SYNONYMS [CAS#]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4].

SAMPLING

SAMPLER: FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm diameter; conductive cassette)

FLOW RATE: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)
*Adjust for 100 to 1300 fibers/mm^2

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): see EVALUATION OF METHOD

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: MICROSCOPY, TRANSMISSION ELECTRON (TEM)

ANALYTE: asbestos fibers

SAMPLE PREPARATION: modified Jaffe wick

EQUIPMENT: transmission electron microscope; energy dispersive X-ray system (EDX) analyzer

CALIBRATION: qualitative electron diffraction; calibration of TEM magnification and EDX system

RANGE: 100 to 1300 fibers/mm^2 filter area [1]

ESTIMATED LOD: 1 confirmed asbestos fiber above 95% of expected mean blank value

PRECISION ($\hat{S}_r$): 0.28 when 65% of fibers are asbestos; 0.20 when adjusted fiber count is applied to PCM count [2].

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. This method is use d to determine asbestos fibers in the optically visible range and is intended to complement the results obtained by phase con trast microscopy (Method 7400).

INTERFERENCES: Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification. Some non-asbestos amphibole m inerals may give electron diffraction patterns similar to asbestos amphiboles.

OTHER METHODS: This method is designed for use with Method 7400 (phase contrast microscopy).

REAGENTS:

1. Acetone. (See SPECIAL PRECAUTIONS.)

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl, cellulose ester membrane filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean count is >5 fibers/100 fields. These are defined as laboratory blanks.
   NOTE 2: Use an electrically-conductive extension cowl to reduce electrostatic effects on fiber sampling and during sample shipment. Ground the cowl when possible during sampling.
   NOTE 3: 0.8-µm pore size filters are recommended for personal sampling. 0.45-µm filters are recommended for sampling when performing TEM analysis on the samples because the particles deposit closer to the filter surface. However, the higher pressure drop through these filters normally preclude their use with personal sampling pumps.
2. Personal sampling pump, 0.5 to 16 L/min, with flexible connecting tubing.
3. Microscope, transmission electron, operated at ca. 100 kV, with electron diffraction and energy-dispersive X-ray capabilities, and having a fluorescent screen with inscribed or overlaid calibrated scale (Step 15).
   NOTE: The scale is most efficient if it consists of a series of lines inscribed on the screen or partial circles every 2 cm distant from the center.
4. Diffraction grating replica with known number of lines/mm.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Knife, surgical steel, curved-blade.
7. Tweezers.
8. Grids, 200-mesh TEM copper, (optional: carbon-coated).
9. Petri dishes, 15-mm depth. The top and bottom of the petri dish must fit snugly together. To assure a tight fit, grind the top and bottom pieces together with an abrasive such as carborundum to produce a ground-glass contact surface.
10. Foam, clean polyurethane, spongy, 12-mm thick.
11. Filters, Whatman No. 1 qualitative paper or equivalent, or lens paper.
12. Vacuum evaporator.
13. Cork borer, (about 8-mm).
14. Pen, waterproof, marking.
15. Reinforcement, page, gummed.
16. Asbestos standard bulk materials for reference; e.g. SRM #1866, available from the National Institute of Standards and Technology.
17. Carbon rods, sharpened to 1 mm x 8 mm.
18. Microscope, light, phase contrast (PCM), with Walton-Beckett graticule (see method 7400).
19. Grounding wire, 22-gauge, multi-strand.
20. Tape, shrink- or adhesive-.

SPECIAL PRECAUTIONS: Acetone is extremely flammable (flash point = 0 °F). Take precautions not to ignite it. Heating of acetone must be done in a fume hood using a flameless, spark-free heat source. Asbestos is a confirmed human carcinogen. Handle only in a well-ventilated fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. For personal sampling, fasten sampler to worker's lapel near worker's mouth. Remove the top cover from cowl extension ("open-face") and orient sampler face down. Wrap joint between extender and monitor body with tape to help hold the cassette together and provide a marking surface to identify the cassette. Where possible, especially at low %RH, attach sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove top covers from the field blank cassettes and store top covers and cassettes in a clean area (e.g., closed bag or box) during sampling. Replace top covers when sampling is completed.
4. Sample at 0.5 to 16 L/min [3]. Adjust sampling rate, Q (L/min), and time, t (min), to produce fiber density, E, of 100 to 1300 fibers/mm ^{2} [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area (A_c = 385 mm^2)] for optimum accuracy. Do not exceed ca. 0.5 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

   NOTE: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 h (700 to 2800 L) is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust [3].
5. At the end of sampling, replace top cover and small end caps.
6. Ship samples upright with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in the shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

7. Remove circular sections from any of three quadrants of each sample and blank filter using a cork borer [4]. The use of three grid preparations reduces the effect of local variations in dust deposit on the filter.
8. Affix the circular filter sections to a clean glass slide with a gummed page reinforcement. Label the slide with a waterproof marking pen.
   NOTE: Up to eight filter sections may be attached to the same slide.
9. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 min for the sample filter(s) to fuse and clear.
   NOTE: The "hot block" clearing technique [5] of Method 7400 or the DMF clearing technique [6] may be used instead of steps 8 and 9.
10. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1-by 5-mm section of a graphite rod onto the cleared filter(s). Remove the slide to a clean, dry, covered petri dish [4].
11. Prepare a second petri dish as a Jaffe wick washer with the wicking substrate prepared from filter or lens paper placed on top of a 12-mm thick disk of clean, spongy polyurethane foam [7].

Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent.

NOTE: The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

12. Place the TEM grid on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish halves and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated.

NOTE: The level of acetone should be just high enough to saturate the filter paper without creating puddles.

13. Remove about a quarter section of the carbon-coated filter from the glass slide using a surgical knife and tweezers. Carefully place the excised filter, carbon side down, on the appropriately-labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to raise the acetone level as high as possible without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it (allowing drops of condensed acetone to form near the edge rather than in the center where they would drip onto the grid preparation).

CALIBRATION AND QUALITY CONTROL:

14. Determine the TEM magnification on the fluorescent screen:
    a. Define a field of view on the fluorescent screen either by markings or physical boundaries.
       NOTE: The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric) [7].
    b. Insert a diffraction grating replica into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that goniometer stage tilt is zero.
    c. Adjust microscope magnification to 10,000X. Measure the distance (mm) between the same relative positions (e.g., between left edges) of two widely-separated lines on the grating replica. Count the number of spaces between the lines.
       NOTE: On most microscopes the magnification is substantially constant only within the central 8- to 10-cm diameter region of the fluorescent screen.
    d. Calculate the true magnification (M) on the fluorescent screen:

$$m = \frac{X \cdot G}{Y}$$

       where: X = total distance (mm) between the two grating lines;
              G = calibration constant of the grating replica (lines/mm);
              Y = number of grating replica spaces counted
    e. After calibration, note the apparent sizes of 0.25 and 5.0 µm on the fluorescent screen. (These dimensions are the boundary limits for counting asbestos fibers by phase contrast microscopy.)
15. Measure 20 grid openings at random on a 200-mesh copper grid by placing a grid on a glass slide and examining it under the PCM. Use the Walton-Beckett graticule to measure the grid opening dimensions. Calculate an average graticule field dimension from the data and use this number to calculate the graticule field area for an average grid opening.
    NOTE: A grid opening is considered as one graticule field.
16. Obtain reference selected area electron diffraction (SAED) or microdiffraction patterns from standard asbestos materials prepared for TEM analysis.
    NOTE: This is a visual reference technique. No quantitative SAED analysis is required [7]. Microdiffraction may produce clearer patterns on very small fibers or fibers partially obscured by other material.
    a. Set the specimen holder at zero tilt.

b. Center a fiber, focus, and center the smallest field-limiting aperture on the fiber. Obtain a diffraction pattern. Photograph each distinctive pattern and keep the photo for comparison to unknowns.

NOTE: Not all fibers will present diffraction patterns. The objective lens current may need adjustment to give optimum pattern visibility. There are many more amphiboles which give diffraction patterns similar to the analytes named on p. 7402-1. Some, but not all, of these can be eliminated by chemical separations. Also, some non-amphiboles (e.g., pyroxenes, some talc fibers) may interfere.

17. Acquire energy-dispersive X-ray (EDX) spectra on approximately 5 fibers having diameters between 0.25 and 0.5 µm of each asbestos variety obtained from standard reference materials [7].

NOTE: The sample may require tilting to obtain adequate signal. Use same tilt angle for all spectra.

a. Prepare TEM grids of all asbestos varieties.
b. Use acquisition times (at least 100 sec) sufficient to show a silicon peak at least 75% of the monitor screen height at a vertical scale of ≥500 counts per channel.
c. Estimate the elemental peak heights visually as follows:
   (1) Normalize all peaks to silicon (assigned an arbitrary value of 10).
   (2) Visually interpret all other peaks present and assign values relative to the silicon peak.
   (3) Determine an elemental profile for the fiber using the elements Na, Mg, Si, Ca, and Fe. Example: 0-4-10-3-<1 [7].

   NOTE: In fibers other than asbestos, determination of Al, K, Ti, S, P, and F may also be required for fiber characterization.

   (4) Determine a typical range of profiles for each asbestos variety and record the profiles for comparison to unknowns.

MEASUREMENT:

18. Perform a diffraction pattern inspection on all sample fibers counted under the TEM, using the procedures given in step 17. Assign the diffraction pattern to one of the following structures:
    a. chrysotile;
    b. amphibole;
    c. ambiguous;
    d. none.

    NOTE: There are some crystalline substances which exhibit diffraction patterns similar to those of asbestos fibers. Many of these, (brucite, halloysite, etc.) can be eliminated from consideration by chemistry. There are, however, several minerals (e.g., pyroxenes, massive amphiboles, and talc fibers) which are chemically similar to asbestos and can be considered interferences. The presence of these substances may warrant the use of more powerful diffraction pattern analysis before positive identification can be made. If interferences are suspected, morphology can play an important role in making positive identification.

19. Obtain EDX spectra in either the TEM or STEM modes from fibers on field samples using the procedure of step 18. Using the diffraction pattern and EDX spectrum, classify the fiber:
    a. For a chrysotile structure, obtain EDX spectra on the first five fibers and one out of ten thereafter. Label the range profiles from 0-5-10-0-0 to 0-10-10-0-0 as "chrysotile."
    b. For an amphibole structure, obtain EDX spectra on the first 10 fibers and one out of ten thereafter. Label profiles ca. 0-2-10-0-7 as "possible amosite"; profiles ca. 1-1-10-0-6 as "possible crocidolite"; profiles ca. 0-4-10-3-<1 as "possible tremolite"; and profiles ca. 0-3-10-0-1 as "possible anthophyllite."

       NOTE: The range of profiles for the amphiboles will vary up to ± 1 unit for each of the elements present according to the relative detector efficiency of the spectrometer.

    c. For an ambiguous structure, obtain EDX spectra on all fibers. Label profiles similar to the chrysotile profile as "possible chrysotile." Label profiles similar to the various amphiboles as "possible amphiboles." Label all others as "unknown" or "non-asbestos."

20. Counting and Sizing:
    a. Insert the sample grid into the specimen grid holder and scan the grid at zero tilt at low magnification (ca. 300 to 500X). Ensure that the carbon film is intact and unbroken over ca. 75% of the grid openings.
    b. In order to determine how the grids should be sampled, estimate the number of fibers per grid opening during a low-magnification scan (500 to 1000X). This will allow the analyst to cover most of the area of the grids during the fiber count and analysis. Use the following rules when picking grid openings to count [7,8]:
        (1) Light loading (<5 fibers per grid opening): count total of 40 grid openings.
        (2) Moderate loading (5 to 25 fibers per grid opening): count minimum of 40 grid openings or 100 fibers.
        (3) Heavy loading (>25 fibers per opening): count a minimum of 100 fibers and at least 6 grid openings.

        Note that these grid openings should be selected approximately equally among the three grid preparations and as randomly as possible from each grid.
    c. Count only grid openings that have the carbon film intact. At 500 to 1000X magnification, begin counting at one end of the grid and systematically traverse the grid by rows, reversing direction at row ends. Select the number of fields per traverse based on the loading indicated in the initial scan. Count at least 2 field blanks per sample set to document possible contamination of the samples. Count fibers using the following rules:
        (1) Count all particles with diameter greater than 0.25 µm that meet the definition of a fiber (aspect ratio ≥3:1, longer than 5 µm). Use the guideline of counting all fibers that would have been counted under phase contrast light microscopy (Method 7400). Use higher magnification (10000X) to determine fiber dimensions and countability under the acceptance criteria. Analyze a minimum of 10% of the fibers, and at least 3 asbestos fibers, by EDX and SAED to confirm the presence of asbestos. Fibers of similar morphology under high magnification can be identified as asbestos without SAED. Particles which are of questionable morphology should be analyzed by SAED and EDX to aid in identification.
        (2) Count fibers which are partially obscured by the grid as half fibers.
            NOTE: If a fiber is partially obscured by the grid bar at the edge of the field of view, count it as a half fiber only if more than 2.5 µm of fiber is visible.
        (3) Size each fiber as it is counted and record the diameter and length:
            (a) Move the fiber to the center of the screen. Read the length of the fiber directly from the scale on the screen.
                NOTE 1: Data can be recorded directly off the screen in µm and later converted to µm by computer.
                NOTE 2: For fibers which extend beyond the field of view, the fiber must be moved and superimposed upon the scale until its entire length has been measured.
            (b) When a fiber has been sized, return to the lower magnification and continue the traverse of the grid area to the next fiber.
    d. Record the following fiber counts:
        (1) f_s, f_b = number of asbestos fibers in the grid openings analyzed on the sample filter and corresponding field blank, respectively.
        (2) F_s, F_b = number of fibers, regardless of identification, in the grid openings analyzed on the sample filter and corresponding field blank, respectively.

CALCULATIONS:

21. Calculate and report the fraction of optically visible asbestos fibers on the filter, $(f_s - f_b)/(F_s - F_b)$. Apply this fraction to fiber counts obtained by PCM on the same filter or on other filters for which the TEM sample is representative. The final result is an asbestos fiber count. The type of asbestos present should also be reported.
22. As an integral part of the report, give the model and manufacturer of the TEM as well as the model and manufacturer of the EDX system.

EVALUATION OF METHOD:

The TEM method, using the direct count of asbestos fibers, has been shown to have a precision of 0.275 (s_r) in an evaluation of mixed amosite and wollastonite fibers. The estimate of the asbestos fraction, however, had a precision of 0.11 (s_r). When this fraction was applied to the PCM count, the overall precision of the combined analysis was 0.20 [2].

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METHOD REVISED BY:

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Designation: D5755 – 09

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading[1]

This standard is issued under the fixed designation D5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the surface loading of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the surface loading of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D3195 Practice for Rotameter Calibration

D3670 Guide for Determination of Precision and Bias of Methods of Committee D22

D5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Surface Loading

D6620 Practice for Asbestos Detection Limit Based on Counts

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (1),[3] (2) and (3).

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No.[4]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of <1 mm.

3.2.6 *fiber*—a structure having a minimum length of 0.5 µm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of non-fibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the surface loading of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, no relationship has been established between asbestos-containing dust as measured by this test method and potential human exposure to airborne asbestos. Accordingly, the users should consider other available information in their interpretation of the data obtained from this test method.

5.2 This definition of dust accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos surface loading results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and that a minimum of three samples be analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the

[3] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[4] The non-asbestiform variations of the minerals indicated in 5.1.3 have different Chemical Abstract Service (CAS) numbers.

laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite*.
6.1.2 *Palygorskite (Attapulgite)*.
6.1.3 *Halloysite*.
6.1.4 *Pyroxenes*.
6.1.5 *Sepiolite*.
6.1.6 *Vermiculite scrolls*.
6.1.7 *Fibrous talc*.
6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[5]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA)*.
7.4 *High Vacuum Carbon Evaporator*, with rotating stage.
7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.
7.6 *Exhaust or Fume Hood*.
7.7 *Particle-free Water* (ASTM Type II, see Specification D1193).
7.8 *Glass Beakers* (50 mL).
7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).
7.10 *Waterproof Markers*.
7.11 *Forceps* (tweezers).
7.12 *Ultrasonic Bath*, table top model (100 W).
7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.
7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.
7.15 *Side Arm Filter Flask*, 1000 mL.
7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.
7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤0.2 µm pore size.
7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).
7.19 *Glass Slides*, approximately 76 by 25 mm in size.
7.20 *Scalpel Blades*, No. 10, or equivalent.
7.21 *Cabinet-type Desiccator*, or low temperature drying oven.
7.22 *Chloroform*, reagent grade.
7.23 *Acetone*, reagent grade.
7.24 *Dimethylformamide (DMF)*.
7.25 *Glacial Acetic Acid*.
7.26 *1-methyl-2-pyrrolidone*.
7.27 *Plasma Asher*, low temperature.
7.28 *pH Paper*.
7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.
7.30 *Rotameter*.
7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8µ m or smaller pore size MCE or PC filters.
7.32 *Cork Borer*, 7 mm.
7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.
7.34 *Asbestos Standards*, as outlined in 3.1.2.
7.35 *Tygon*[6] *Tubing*, or equivalent.
7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.
7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.
7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.
7.39 *Copper TEM Finder Grids*, 200 mesh.
7.40 *Carbon Evaporator Rods*.
7.41 *Lens Tissue*.
7.42 *Ashless Filter Paper Filters*, 90 mm diameter.
7.43 *Gummed Paper Reinforcement Rings*.
7.44 *Wash Bottles*, plastic.
7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).
7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).
7.47 *Diffraction Grating Replica*.

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

[5] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[6] Tygon is a registered trademark of the DuPont Co.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D3195 and 7.29).

8.4 Record all calibration information (**5**).

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic “bubble pack” is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank surface loadings (see Section 11).

10.4 All sample preparation steps 10.4.1-10.4.6 shall take place in the dust preparation area inside a HEPA hood.

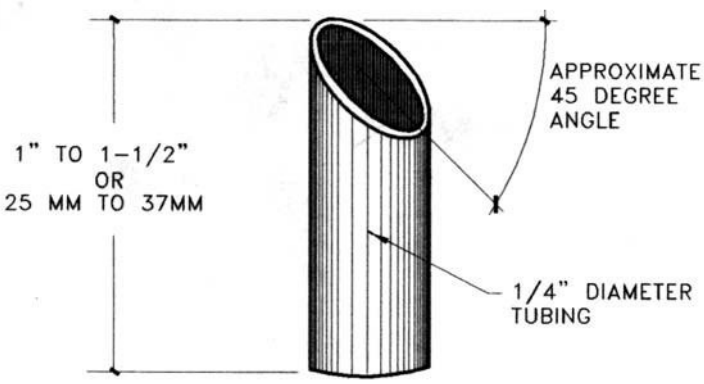

FIG. 1 Example of the Tubing Nozzle

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust solution by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of suspension in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate solution such as diluted India ink (solution of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 µ m pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 µm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing surface loading, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual solution from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the suspension in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8-10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of suspension has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original suspension or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample suspension with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the

previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method*:

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (**4**). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method*:

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (**4**) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (**6**) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts,

separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*— The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (**7, 8**). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer*:

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

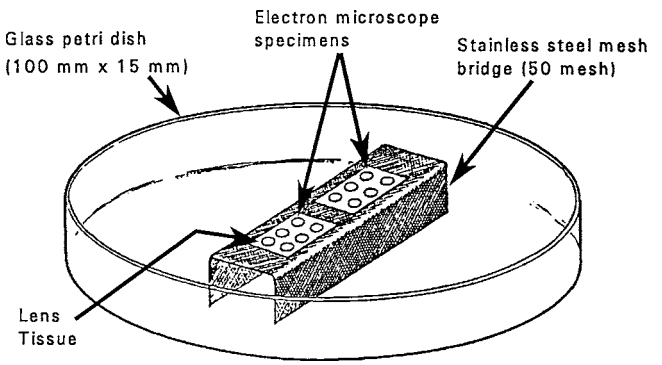

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2—**Caution:** Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 µm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability*:

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the *x* and *y* translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability*:

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing Materials in Schools* document (**4**). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (**7**) or Chatfield and Dillon (**8**).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules*:

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity (AS)*—As determined by the following equation:

$$(EFA \times 100\,mL \times 1)/(GO \times GOA \times V \times SPL) = AS \qquad (1)$$

where:

- EFA = effective filter area of the final sampling filter, mm^2
- GO = number of grid openings counted
- GOA = average grid opening area, mm^2
- SPL = surface area sampled, cm^2
- V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL
- AS = analytical sensitivity, expressed as asbestos structures/cm^2

16.8.1 An AS of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, increasing the area of the collection, or decreasing the size of the final filter. For example, using a collection area = 500 cm^2, filter area = 1000 mm^2, number of grid openings = 10, and a grid area of 0.01 mm^2, V = 50 mL, the AS is 40 str/cm^2. Occasionally, due to high particle loadings or high asbestos surface loading, this AS cannot be practically achieved and stopping rules apply.

16.8.2 The numerical value of AS required for any specific application of this method may be achieved by selecting an appropriate combination of the sampling and analysis parameters in the above equation. For example, if SPL = 100 cm^2, EFA = 1000 mm^2, GO = 10, GOA = 0.01 mm^2, V = 10 mL, and D = 1, then AS = 1000 str/cm^2. Increasing GO to 50 and V to 50 mL changes the AS to 40 Str/cm^2.

16.8.3 When sample filters are heavily loaded with particulate matter, it may useful to employ serial dilutions during

preparation to reduce the loading on grid specimens to acceptable levels and thus facilitate analysis. Under such circumstances, the AS may be calculated by substituting an appropriate value for the dilution factor, D, into the above equation. In general:

$$D = VA/(V + VPFW) \quad (2)$$

VA = the volume of the aliquot from the new, diluted suspension that is filtered to prepare the analytical filter; V = the volume of the aliquot from the initial (100 mL) suspension that is diluted; and VPFW = the volume of particle free water added to V during serial dilution to produce the new, diluted suspension.

Thus, if GO = 10, V = 10 mL, VPFW = 90 mL, and VA = 1.0 mL, D = 0.01 and the AS = 100 000 str/cm^2.

16.9 *Limit of Detection*—The limit of detection for this test method is calculated using the Practice D6620. All data shall be provided in the laboratory report.

16.10 *Stopping Rules*:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an AS of less than 1000 asbestos structures per square centimetre.

16.10.2 If an AS of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Surface loading in structures/cm^2.

18.1.2 The AS.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ \text{mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \quad (3)$$

where:

#STR = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document **(4)** and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document **(6)**. These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM*:

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis

are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(a) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) Frequency of calibration will depend on the service history of the particular microscope.

(c) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(b) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA*:

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \quad (4)$$

where:

4.185 = Joules/cal,
R = energy deposition, watts/mL,
θ_1 = temperature rise with the ultrasonic bath not operating, °C,
θ_2 = temperature rise with the ultrasonic bath operating, °C,
t = time in seconds, 60 s (20.5.3 and 20.5.5),
σ = specific heat of the liquid in the glass sample container, 1.0 cal/g, and
ρ = density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m 3, as defined by this procedure.

21. Precision and Bias [7]

21.1 *Precision*—The precision of this test method is based on an interlaboratory study conducted in 2003. Each of the ten laboratories tested a single material. Every "test result" represents an individual determination. Each laboratory reported duplicate test results for the analyses. Practice E691 was followed for the design and analysis of the data.

21.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material; *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

21.1.1.1 Repeatability limits are listed in Table 1.

21.1.2 *Reproducibility (R)*—Two test results shall be judged not equivalent if they differ by more than the *R* value for that material; *R* is the interval representing the critical difference

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D22-1032.

TABLE 1 Asbestos Structures per cm^2(× 1000)

Material	Average[A] $\overline{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limits *r*	Reproducibility Limits *R*
A	147.80	22.07	85.46	61.80	239.30

[A] The average of the laboratories' calculated averages.

between two test results for the same material, obtained by different operators using different equipment in different laboratories.

21.1.2.1 Reproducibility limits are listed in Table 1.

21.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

21.1.4 Any judgment in accordance with statements 21.1.1 and 21.1.2 would have an approximate 95 % probability of being correct.

21.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

21.3 The precision statement was determined through statistical examination of 20 results, from ten laboratories, on a single type of material, described below.

21.3.1 *Material A*—A chrysotile asbestos fiber-containing dust in a microvacuum cassette. The dust cassettes were prepared by resuspending a sample of World Trade Center dust and allowing it to settle. Samples of the dust from 100-cm^2 areas were collected using a microvacuum cassette following the procedures described in this test method.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client:		Accelerating Voltage:	
Sample ID:		Indicated Mag:	KX
Job Number:		Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:		Filter Type:	
Grid Accepted, 600X:	Yes No	Filter Size:	
Percent Loading:	%	Filter Pore Size (μm):	
Grid Box #1:		Grid Opening:	1) μm x μm
			2) μm x μm

Analyst: _____

Reviewer: _____ Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm^2:	(EFA)	
Number of Grid Openings Counted:	(GO)	
Average Grid Opening Area in mm^2:	(GOA)	
Volume of sample Filtered in ml:	(V)	
Surface area Sampled in cm^2:	(SPL)	
Number of Asbestos Structures Counted:*	(#STR)	

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM^2:

$$\frac{EFAX \times 100 \times \#STR}{GO \times GOA \times VX SPL} = \text{(Asbestos Structures per } cm^2\text{)}$$

Results for Total Asbestos Structures: _____ (Structures per cm^2)

Results for Structures ≥ microns: _____ (Structures per cm^2)

FIG. X1.1 Dust Sample Analysis Work Sheet

Job Number: _____

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: Keys to Abbreviations Used in Figure:

Type:

C	=	Chrysotile
AM	=	Amosite
CR	=	Crocidolite
AC	=	Actinolite
TR	=	Tremolite
AN	=	Anthophyllite
N	=	Non Asbestos

Structure:

F	=	Fiber
B	=	Bundle
C	=	Cluster
M	=	Matrix

Others:

NSD	=	No Structures Detected
Morph	=	Morphology
SAED	=	Selected Area Electron Diffraction
EDS	=	Energy Dispersive X-Ray Spectroscopy
ER	=	Inter-Row Spacing
NP	=	No Pattern

FIG. X1.2 TEM Count Sheet

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